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Woodward-Clyde Consultants

14 November 1990
87X4660

REC'D

11-16-90

FB

Mr Frank Battaglia
USEPA Region I
Waste Management Division
90 Canal Street
Boston, MA 02114

EPA COMMENTS INCORPORATED
IN 1-16-91 FINAL REVISION
DOCUMENT

Dear Frank:

Please find enclosed the revised Data Validation Appendix IX checklists for your final review, plus the Data Summary Forms to be utilized during the Remedial Investigation data validation/data reduction process.

If you have any questions on the above, please do not hesitate to contact Diana Baldi at 919-632-6000 or myself at 201-785-0700, Extension 372.

Very truly yours,

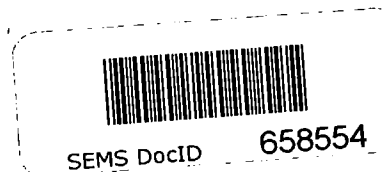


John P. Lorenzo
Senior Staff Scientist/Project Chemist

JPL:ef

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cc: Diana Baldi (Ciba Geigy)
Joanna Hall (Alliance Corp)
Mark Houlday (WCC)



CIBA-GEIGY
INORGANIC REGION I WORKSHEETS
RE-EDITED FOR APPENDIX IX
CONSTITUENTS

Prepared by:

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GREENSBORO, NORTH CAROLINA 27419

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CIBA-GEIGY
INORGANIC REGION I WORKSHEET

Background: The term hazardous constituent used in the Solid Waste Disposal Act Section 3004(u) means constituents found in Appendix VIII of 40CFR part 261. EPA also defines hazardous constituents as those constituents identified in Appendix IX of 40CFR part 264. Appendix IX constituents generally constitutes a subset of Appendix VIII which are particularly suitable for ground water analyses.

In general, where very little is known of waste characteristics, and where there is a potential for a wide spectrum of wastes to have been released, only then is an owner/operator required to perform an extensive routine analysis for a broader spectrum of waste such as an Appendix IX analysis.

Radian Corporation of Austin, Texas, has been sub-contracted by WCC to analyze the 19 inorganic hazardous constituents in Appendix IX and will be utilizing the following SW-846 procedures listed in Table 1. As such, the enclosed Inorganic Region I Data Validation Worksheets have been modified accordingly for each fraction to conform to the QA/QC criteria of each SW-846 test methods in Table 1.

TABLE 1
SELECTED ANALYTICAL METHODS FOR
INORGANIC APPENDIX IX ANALYSES

SW-846 Method	General Category/ Analyte	Technique	Number of Analytes Measured
6010	Metals	ICP	11
7041	Antimony	GFAA	1
7060	Arsenic	GFAA	1
7421	Lead	GFAA	1
7470	Mercury	CVAA	1
7740	Selenium	GFAA	1
7841	Thallium	GFAA	1
9012	Cyanide	Colorimetric	1
9030	Sulfide	Titrimetric	1
TOTAL			19

- ICP - Inductively Coupled Plasma Spectrometry (all other metals)
 GFAA - Graphite Furnace Atomic Absorption Spectrometry (normally only arsenic, antimony, lead, selenium, and thallium)
 CVAA - Cold Vapor Atomic Absorption Spectroscopy (mercury analyses only)

INORGANIC METALS
SUMMARY TABLES

NON-FILTERED SAMPLES

Case #: _____ Sampling Date(s): _____

**+Due to dilution, sample quantitation limit is affected.
See dilution table for specifics.**

SEE NARRATIVE FOR CODE DEFINITIONS
revised 10/90

DATA SUMMARY FORM: I N O R G A N I C S

WATER SAMPLES (DISSOLVED)

ALTERED SAMPLES

Case #: _____ **Sampling Date(s):** _____

+Due to dilution, sample quantitation limit is affected.
See dilution table for specifics.

~~PQL = Contract Required Detection Limit~~
PRACTICAL QUANTITATION

***Action Level Exists**

SEE NARRATIVE FOR CODE DEFINITIONS
revised 10/90

APPENDIX II METALS
DATA SUMMARY FORM: INORGANICS

Page ____ of ____

Site Name: _____
Case #: _____ Sampling Date(s): _____

SOIL SAMPLES
(ug/kg) PPM

+Due to dilution, sample quantitation limit is affected.
See dilution table for specifics.

Sample No. Dilution Factor Location													
PQL	ANALYTE	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q
3	ANTIMONY												
0.2	*Arsenic												
1.0	Barium												
0.2	Beryllium												
0.5	*Cadmium												
1.0	*Chromium												
2	Cobalt												
2	Copper												
0.2	*Lead												
0.2	Mercury												
2	*Nickel												
0.2	Selenium												
1.0	Silver												
10	TIN												
0.2	Thallium												
2	Vanadium												
2	Zinc												

PQL = Contract Required Detection Limit
PRACTICAL QUANTITATION

*Action Level Exists

SEE NARRATIVE FOR CODE DEFINITIONS
revised 10/90

REGION I

Site Name _____
Reference Number _____

REGION I REVIEW OF INORGANIC
SW-846 (3RD EDITION)

The hardcopied (laboratory name) _____ data package received at Region I has been reviewed and the quality assurance and performance data summarized. The data review included:

Matrix:

SDG. No.:

No. of Samples:

Sample Identifiers:

Trip Blank No.:

Equipment Blank No.:

Field Dup Nos.:

Sampling Date(s):

Shipping Date(s):

Date Rec'd by Lab:

SW-846 (3rd Edition) requires that specific analytical work be done. The general criteria used to determine the performance were based on an examination of:

-Data Completeness

-Holding Times

-Calibrations

-Blanks

-ICP Interference Check Results

-Matrix Spike Recoveries

-Laboratory Duplicates

-Field Duplicates

-Lab Control Sample Results

-Furnace AA Results

-ICP Serial Dilution Results

-Detection Limit Results

-Sample Quantitation

Overall Comments:

Definitions and Qualifiers:

A - Acceptable data.

J - Approximate data due to quality control criteria.

R - Reject data due to quality control criteria

U - Analyte not detected

Reviewer:

Date:

REGION I
Data Review Worksheets

I. DATA COMPLETENESS

MISSING INFORMATION

DATE LAB CONTACTED

DATE REC'D

REGION I
Data Review Worksheets

II. HOLDING TIMES

Complete table for all samples and circle the analysis date for samples not within criteria.

SAMPLE ID	DATE SAMPLED	HG DATE ANALYSIS	CYANIDE DATE ANALYSIS	OTHERS DATE ANALYSIS	pH	ACTION
--------------	-----------------	------------------------	-----------------------------	----------------------------	----	--------

METALS - 180 DAYS FROM SAMPLE COLLECTION
MERCURY - 28 DAYS FROM SAMPLE COLLECTION
CYANIDE - 14 DAYS FROM SAMPLE COLLECTION

ACTION:

1. If holding times are exceeded all positive results are estimated (J) and non-detects are estimated (UJ).
2. If holding times are grossly exceeded, the reviewer may determine that non-detects are unusable (R).

REGION I
Data Review Worksheets

III A. INSTRUMENT CALIBRATION (Section 1)

1. Recovery Criteria

List the analytes which did not meet the percent recovery (%R) criteria for Initial or Continuing Calibration.

<u>DATE</u>	<u>ICV/CCV#</u>	<u>ANALYTE</u>	<u>%R</u>	<u>ACTION</u>	<u>SAMPLES AFFECTED</u>
-------------	-----------------	----------------	-----------	---------------	-------------------------

ACTIONS:

If any analyte does not meet the %R criteria follow the actions stated below:

For Positive Results:

	<u>Accept</u>	<u>Estimate (J)</u>	<u>Reject (R)</u>
Metals	90-110%R	75-89%R, 111-125%R	<75%R, >125%R
Mercury	80-120%R	65-79%R, 121-135%R	<65%R, >135%R
Cyanide	85-115%R	70-84%R, 116-130%R	<70%R, >130%R

For Non-detected Results:

	<u>Accept</u>	<u>Estimate (UJ)</u>	<u>Reject (R)</u>
Metals	90-125%R	75-89%R	<75%R, >125%R
Mercury	80-135%R	65-79%R	<65%R, >135%R
Cyanide	85-130%R	70-84%R	<70%R, >130%R

REGION I
Data Review Worksheets

III B. INSTRUMENT CALIBRATION (Section 2)

2. Analytical Sequence

- | | | |
|----|---|-----------|
| A. | Did the laboratory use the proper number of standards for calibration as described in the SW-846 (3rd Edition) manual. There is no difference on the proper number of standards between the CLP-SOW and SW-846. | Yes or No |
| | | |
| B. | Were calibrations performed at the beginning of each day (or every 8 hours): whichever is more frequent. | Yes or No |
| | | |
| C. | Were midpoint calibration standards analyzed at the beginning of sample analysis and at a minimum frequency of ten percent? | Yes or No |
| | | |
| D. | Were the correlation coefficients for the calibration curves for AA, Hg, and CN ≤ 0.995 ? | Yes or No |

If No,

The data may be affected. Use professional judgement to determine the severity of the effect and qualify the data accordingly. Discuss any actions below and list the samples affected.

REGION I
Data Review Worksheet

IV A. BLANK ANALYSIS RESULTS (Sections 1-3)

List the blank contamination in Sections 1 & 2 below. A separate worksheet must be used for soil and water blanks.

MATRIX: _____

1. Laboratory Blanks

<u>DATE</u>	<u>ICB/CCB#</u>	<u>PREP BL</u>	<u>ANALYTE</u>	<u>CONC./UNITS</u>
-------------	-----------------	----------------	----------------	--------------------

2. Equipment/Trip Blanks

<u>DATE</u>	<u>EQUIP BL#</u>	<u>ANALYTE</u>	<u>CONC./UNITS</u>
-------------	------------------	----------------	--------------------

3. Frequency Requirements

- | | | |
|----|--|-----------|
| A. | Was a preparation blank carried through the entire analytical process for each matrix type or group of 20 samples, whichever is more frequent? | Yes or No |
| B. | Was a reagent blank with a minimum of three standards run daily? | Yes or No |
| C. | If 20 or more samples were run, was a reagent blank analyzed along with a mid-range standard at a frequency of every 10 samples? | Yes or No |

If No,

The data may be affected. Use professional judgement to determine the severity of the effect and qualify the data accordingly. Discuss any actions below, and list the samples affected.

REGION I
Data Review Worksheets

IV B. BLANK ANALYSIS RESULTS (Section 4)

4. Blank Actions

The Action Levels for any analyte is equal to five times the highest concentration of that element's contamination in any blank. The action level for samples which have been concentrated or diluted should be multiplied by the concentration/dilution factor. No positive sample result should be reported unless the concentration of the analyte in the sample exceeds the Action Level (AL). Specific actions are as follows:

1. When the concentration is greater than the IDL, but less than the Action Level, report the sample concentration detected with a U.
2. When the sample concentration is greater than the Action Level, report the sample concentration unqualified.

MATRIX: _____

MATRIX: _____

<u>ELEMENT</u>	<u>MAX. CONC./</u> <u>UNITS</u>	<u>AL/</u> <u>UNITS</u>
----------------	------------------------------------	----------------------------

<u>ELEMENT</u>	<u>MAX. CONC./</u> <u>UNITS</u>	<u>AL/</u> <u>UNITS</u>
----------------	------------------------------------	----------------------------

NOTE: Blanks analyzed during a soil case must be converted to mg/kg in order to compare them with the sample results.

$$\text{conc. in ug/l} \times \frac{\text{volume diluted to (100 ml)}}{\text{weight digested (1 gram)}} \times \frac{1 \text{ liter}}{1000 \text{ ml}} \times \frac{1000 \text{ gms.}}{1 \text{ kg}} \times \frac{1 \text{ mg}}{1000 \text{ ug}} = \frac{\text{mg}}{\text{kg}}$$

Multiplying this result by 5 to arrive at the action level gives a final result in mg/kg which can then be compared to sample results.

The SW-846 (3rd Edition) requirement is that the calibration blank be within a three (3) standard deviation window of the mean blank value. As such, gross blank contamination warrants the data validator to contact the laboratory to verify this was performed. List all anomalies in the Inorganic Regional Data Assessment.

REGION I
Data Review Worksheets

V A. ICP INTERFERENCE CHECK SAMPLE (Sections 1 & 2)

1. Recovery Criteria

List any elements in the ICS AB solution which did not meet the criteria for %R. SW-846 (3rd Edition) does warrant a $\pm 20\%$ window of the true value. The laboratory in accordance with SW-846 Method 6010 must follow an established control limit of 1.5 times the standard deviation of the mean value. If reoccurring problems arise, contact the lab and determine if any deviation from this procedure has occurred.

<u>DATE</u>	<u>ELEMENT</u>	<u>%R</u>	<u>ACTION</u>	<u>SAMPLES AFFECTED</u>
-------------	----------------	-----------	---------------	-----------------------------

ACTIONS:

If an element does not meet the %R criteria, follow the actions stated below:

	<u>PERCENT RECOVERY</u>		
	<50%	50-79%	>120%
Positive Sample Results	R	J	J
Non-detected Sample Results	R	UJ	A

2. Frequency Requirements

Were Interference QC samples run at the beginning and end of each batch analysis run or a minimum of twice per 8 hour working shift, whichever is more frequent?

Yes or No

If no,

The data may be affected. Use professional judgement to determine the severity of the effect and qualify the data accordingly. Discuss any actions below and list the samples affected.

REGION I
Data Review Worksheets

V B. ICP INTERFERENCE CHECK SAMPLE (Section 3)

3. Report the concentration of any elements detected in the ICS A solution > 2xIDL that should not be present.

<u>ELEMENT</u>	CONC. DETECTED <u>IN THE ICS</u>	CONC. OF INTERFERENTS IN THE ICS			
		<u>AL</u>	<u>CA</u>	<u>FE</u>	<u>MG</u>

Estimate the concentration produced by the interfering element in all affected samples. See guidelines for examples. List the samples affected by interferences below:

SAMPLE AFFECTED	ELEMENT AFFECTED	SAMPLE CONC. (ug/L)	SAMPLE INTERFERENT CONC.				ESTIMATED INTERF. (ug/L)
			AL	CA	FE	MG	

ACTIONS:

1. In general, the sample data can be accepted without qualification if the sample concentrations of Al, Ca, Fe, and Mg are less than 50% of their respective levels in the ICS solution.
2. Estimate (J) positive results for affected elements for samples with levels of interferences 50% or more of that in the ICS solution.
3. Reject (R) positive results if the reported concentration is due entirely to the interfering element.
4. Estimate (UJ) non-detected results for which false negatives are suspect.

Give explanations for any actions taken below:

REGION I
Data Review Worksheets

VI. MATRIX SPIKE

TR # _____

MATRIX: _____

1. Recovery Criteria

List the percent recoveries for analytes which did not meet the required criteria.

S - amount of spike added
SSR - spikes sample request
SR - sample result

Analyte	SSR	SR	S	%R	Action
---------	-----	----	---	----	--------

Matrix Spike Actions apply to all samples of the same matrix.

ACTIONS:

- If the sample concentration exceeds the spike concentration by a factor of 4 or more, no action is taken.
- If any analyte does not meet the %R criteria follow the actions stated below:

	<u>PERCENT RECOVERY</u>		
	<u><30%</u>	<u>30%-74%</u>	<u>>125%</u>
Positive Sample Results	J	J	J
Non-detected Results	R	UJ	No Action

2. Frequency Criteria

- Was a matrix spike prepared at the required frequency?
Yes or No

A separate worksheet should be used for each matrix spike pair.

VII. LABORATORY DUPLICATES

List the concentrations of any analyte not meeting the criteria for duplicate precision. For soil duplicates, calculate the PQL in mg/kg using the sample weight, volume and percent solids data for the sample. Indicate what criteria was used to evaluate precision by circling either the RPD or PQL for each element.

MATRIX: _____

<u>Element</u>	<u>PQL*</u>		<u>Sample #</u>	<u>Duplicate #</u>	<u>RPD**</u>	<u>Action</u>
	<u>water</u> ug/L	<u>soil</u> mg/kg				
Antimony	30					
Arsenic	2					
Barium	10					
Beryllium	2					
Cadmium	5					
Chromium	10					
Cobalt	20					
Copper	20					
Lead	2					
Mercury	0.2					
Nickel	20					
Selenium	2					
Silver	10					
Thallium	2					
Tin	100					
Vanadium	20					
Zinc	20					
Cyanide	10					

Laboratory Duplicate Actions should be applied to all other samples of the same matrix type.

ACTIONS:

1. Estimate (J) positive results for elements which have an RPD >20% for waters and >35% for soils.
 2. If sample results are less than 5x the PQL, estimate (J) positive results for elements whose absolute difference is >PQL, (2xPQL for soils). If both samples are non-detected, the RPD is not calculated (NC).
- * No specific detection limits are required in SW-846. All PQLs are of a recommended laboratory calculated nature. As such, all CLP-CRDLS will be substituted with the Radian calculated PQLs for reporting purposes.
- ** Mean RPD of $\pm 20\%$ or ± 2 standard deviations of the last 25 runs (whichever is tighter) for all analytes as warranted by SW-846 (3rd Edition) protocols will be utilized.

VIII. FIELD DUPLICATES

List the concentrations of all analytes in the field duplicate pair. For soil duplicates, calculate the PQL in mg/kg using the sample weight, volume and percent solids data for the sample. Indicate what criteria was used to evaluate the precision by circling either the RPD or PQL for each element.

MATRIX: _____

<u>Element</u>	<u>PQL</u>		<u>Sample #</u>	<u>Duplicate #</u>	<u>RPD**</u>	<u>Action</u>
	<u>water</u> ug/L	<u>soil</u> mg/kg				
Antimony	30					
Arsenic	2					
Barium	10					
Beryllium	2					
Cadmium	5					
Chromium	10					
Cobalt	20					
Copper	20					
Lead	2					
Mercury	0.2					
Nickel	20					
Selenium	2					
Silver	10					
Sodium	5000					
Thallium	2					
Tin	100					
Vanadium	20					
Zinc	20					
Cyanide	10					

Field Duplicate actions should be applied to all other samples of the same matrix type.

ACTIONS:

1. Estimate (J) positive results for elements which have an RPD >30% for waters and >50% for soils.
 2. If sample results are less than 5x the PQL, estimate (J) positive results and (UJ) nondetected results for elements whose absolute difference is >2xPQL, (4xPQL for soils). If both samples are non-detected, the RPD is not calculated (NC).
- * No specific detection limits are required in SW-846. All PQLs are of a recommended laboratory calculated nature. As such, all CLP-CRDLs will be substituted with the Radian calculated PQLs for reporting purposes.
- ** Mean RPD of $\pm 20\%$ or ± 2 standard deviations of the last 25 runs (whichever is tighter) for all analytes as warranted based on SW-846 (3rd Edition) protocols will be utilized.

IX. LABORATORY CONTROL SAMPLE

1. Aqueous LCS

List any LCS recoveries not within the 80-120% criteria and the samples affected.

<u>DATE</u>	<u>ELEMENT</u>	<u>%R</u>	<u>ACTION</u>	<u>SAMPLES AFFECTED</u>
-------------	----------------	-----------	---------------	-------------------------

Note: The SW-846 (3rd Edition) LCS recovery window is $\pm 20\%$. The current 80 - 100% window is acceptable.

2. Solid LCS

List any analytes that were not within the control windows set by the EPA manufacturers for the solid LCS sample. Lot specifications are available on request from Radian.

<u>ELEMENT</u>	<u>LCS CONC.</u>	<u>CONTROL WINDOWS</u>	<u>ACTION</u>	<u>SAMPLES AFFECTED</u>
----------------	------------------	------------------------	---------------	-------------------------

ACTIONS:

<u>AQUEOUS LCS</u>	<u><50%</u>	<u>51-79%</u>	<u>>120%</u>
--------------------	----------------	---------------	-----------------

Positive Results	R	J J	
Non-detected Results	R	UJ A	

<u>SOLID LCS</u>	<u><EPA Control Windows</u>	<u>>EPA Control Windows</u>
------------------	--------------------------------	--------------------------------

Positive Results	J J	
Non-detected Results	UJ A	

3. Frequency Criteria

A. Was an LCS analyzed for every matrix, every digestion batch, and every 20 samples?	Yes or No
---	-----------

X A. FURNACE ATOMIC ABSORPTION ANALYSIS

1. Duplicate Precision

Duplicate injections must agree within $\pm 20\%$.

If duplicate injections do not agree within $\pm 20\%$ for samples/elements the laboratory must rerun and report the lowest coefficient of variation as per SW-846 (3rd Edition) protocols.

2. Post Digestion Spike Recoveries

Spike recoveries met the 75-125% recovery criteria for all samples.

Method of Standard Addition (MSA) is not being performed during round I of sampling.
(Refer to Action #3 below).

Note CLP requirements and not SW-846 will be used as guidance when applying the qualification actions below.

ACTIONS:

1. Estimate (J) positive results if duplicate injections are outside $\pm 20\%$ RSD or CV.
2. If the sample absorbance is $<50\%$ of post digestion spike absorbance the following actions should be applied:

	<u>PERCENT RECOVERY</u>		
	<u>$<10\%$</u>	<u>11%-84%</u>	<u>$>115\%$</u>
Positive Sample Results	R	J	J
Non-detected Results	R	UJ	No Action

3. Estimate (J) sample results if MSA was required.

XI. INDUCTIVELY COUPLED PLASMA (ICP) SERIAL DILUTION ANALYSIS

Serial Dilutions were performed for each matrix and results of the diluted sample analysis agreed within ten percent of the original undiluted analysis as per CLP guidance and not SW-846 (3rd Edition) protocols.

Serial Dilutions were not performed for the following:

Serial Dilutions were performed, but analytical results did not agree within 10% for analyte concentrations greater than 50x the IDL before dilution.

Report all results below that do not meet the required laboratory criteria for ICP serial dilution analysis.

MATRIX: _____

ELEMENT	IDL	50xIDL	SAMPLE RESULT	SERIAL DILUTION	%D	ACTION
Barium						
Beryllium						
Cadmium						
Chromium						
Cobalt						
Copper						
Nickel						
Silver						
Sodium						
Tin						
Vanadium						
Zinc						

Actions apply to all samples of the same matrix.

ACTIONS:

1. Estimate (J) positive results if %D > 15.

Note: Sample result must be $\geq 50x$ PQL for calculation by serial dilution; then use $\pm 10\%$ original undiluted value as criteria.

XII. DETECTION LIMIT RESULTS

1. Instrument Detection Limits

Instrument Detection Limit results were present and found to be less than the Contract Required Detection Limits.

IDLs were not included in the data package on Form XI.

IDLs were present, but the criteria was not met for the following elements: _____

2. Reporting Requirements

Were sample results on Form I reported down to the IDL not the CRDL for all analytes?

Yes or No

Were sample results that were analyzed by ICP for Se, Tl, As, or Pb at least 5X IDL.

Yes or No

Were sample weights, volumes, and dilutions taken into account when reporting detection limits on Form I.

Yes or No

If No,

The reported results may be inaccurate. Make the necessary changes on the data summary tables and request that the laboratory resubmit the corrected data.

XIII. SAMPLE QUANTITATION

Sample results fall within the linear range for ICP and within the calibrated range for all other parameters.

Sample results were beyond the linear range/calibration range of the instrument for the following samples/elements:

In the space below, please show a minimum of one sample calculation per method:

Lab formula: $\frac{\text{ug}}{\text{ul}} \times \frac{100 \text{ ml}}{\text{g}} = \text{ug/g (mg/kg)}$

Water $\frac{\text{ug}}{1} \times \frac{100 \text{ ml}}{100 \text{ ml}} = \text{ug/l}$

ICP

FURNACE

MERCURY

CYANIDE

For soil samples, the following equation may be necessary to convert raw data values (usually reported in ug/L) to actual sample concentrations (mg/kg):

The lab is required to use 1 gram sample (wet weight) to 100 ml.

Wet weight concentration =

Solid digest conc. in $\frac{\text{ug}}{\text{L}} \times \frac{200 \text{ ml}}{1 \text{ gm}} \times \frac{\text{IL}}{1000 \text{ ml}} \times \frac{1000 \text{ gm}}{1 \text{ kg}} \times \frac{1 \text{ mg}}{1000 \text{ ug}} = \frac{\text{mg}}{\text{kg}}$

In addition the sample results are converted to dry weight using the percent solids calculations:

$\frac{\text{Wet weight conc.}}{\% \text{solids}} \times 100 = \text{final concentration, dry weight (mg/kg)}$

REFERENCE NO. _____ SITE _____
LABORATORY _____ NO. OF SAMPLES/
MATRIX _____

SDG # _____
REVIEWER (IF NOT ESD) _____
SW-846 _____
REVIEWER'S NAME _____

COMPLETION DATE _____

DATA ASSESSMENT SUMMARY

	ICP	AA	Hg	CYANIDE
1. HOLDING TIMES	_____	_____	_____	_____
2. CALIBRATIONS	_____	_____	_____	_____
3. BLANKS	_____	_____	_____	_____
4. ICS	_____	_____	_____	_____
5. LCS	_____	_____	_____	_____
6. DUPLICATE ANALYSIS	_____	_____	_____	_____
7. MATRIX SPIKE	_____	_____	_____	_____
8. MSA (not performed)	__	__	__	__
9. SERIAL DILUTION	_____	_____	_____	_____
10. SAMPLE VERIFICATION	_____	_____	_____	_____
11. OTHER QC	_____	_____	_____	_____
12. OVERALL ASSESSMENT	_____	_____	_____	_____

O = Data had no problems/or qualified due to minor problems.

M = Data qualified due to major problems.

Z = Data unacceptable.

X = Problems, but do not affect data.

ACTION ITEMS: _____

AREAS OF CONCERN: _____

CIBA-GEIGY
INORGANIC REGION I WORKSHEETS
RE-EDITED FOR APPENDIX IX
CONSTITUENTS

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SW-846 Method	General Category/ Analyte	Technique	Number of Analytes Measured
6010	Metals	ICP	11
7041	Antimony	GFAA	1
7060	Arsenic	GFAA	1
7421	Lead	GFAA	1
7470	Mercury	CVAA	1
7740	Selenium	GFAA	1
7841	Thallium	GFAA	1
9012	Cyanide	Colorimetric	1
9030	Sulfide	Titrimetric	1
TOTAL			19

- ICP - Inductively Coupled Plasma Spectrometry (all other metals)
GFAA - Graphite Furnace Atomic Absorption Spectrometry (normally only arsenic, antimony, lead, selenium, and thallium)
CVAA - Cold Vapor Atomic Absorption Spectroscopy (mercury analyses only)

INORGANIC METALS

SUMMARY TABLES

NON-FILTERED SAMPLES

Case #: Sampling Date(s): _____

**+Due to dilution, sample quantitation limit is affected.
See dilution table for specifics.**

SEE NARRATIVE FOR CODE DEFINITIONS
revised 10/90

DATA SUMMARY FORM: I N O R G A N I C S

Site Name: _____

Case #: **Sampling Date(s):** _____

**+Due to dilution, sample quantitation limit is affected.
See dilution table for specifics.**

[illegible]

PQL = Contract Required Detection Limit
PRACTICAL QUANTITATION

***Action Level Exists**

SEE NARRATIVE FOR CODE DEFINITIONS
revised 10/90

SOIL SAMPLES

(ug/kg) PPM

ase #: _____ Sampling Date(s): _____

**+Due to dilution, sample quantitation limit is affected.
See dilution table for specifics.**

~~PQL = Contract Required Detection Limit~~
PRACTICAL QUANTITATION

***Action Level Exists**

SEE NARRATIVE FOR CODE DEFINITIONS
revised 10/90

REGION I

Site Name _____
Reference Number _____

**REGION I REVIEW OF INORGANIC
SW-846 (3RD EDITION)**

The hardcopied (laboratory name) _____ data package received at Region I has been reviewed and the quality assurance and performance data summarized. The data review included:

Matrix:

SDG. No.:

No. of Samples:

Sample Identifiers:

Trip Blank No.:

Equipment Blank No.:

Field Dup Nos.:

Sampling Date(s):

Shipping Date(s):

Date Rec'd by Lab:

SW-846 (3rd Edition) requires that specific analytical work be done. The general criteria used to determine the performance were based on an examination of:

-Data Completeness

-Holding Times

-Calibrations

-Blanks

-ICP Interference Check Results

-Matrix Spike Recoveries

-Laboratory Duplicates

-Field Duplicates

-Lab Control Sample Results

-Furnace AA Results

-ICP Serial Dilution Results

-Detection Limit Results

-Sample Quantitation

Overall Comments:

Definitions and Qualifiers:

A - Acceptable data.

J - Approximate data due to quality control criteria.

R - Reject data due to quality control criteria

U - Analyte not detected

Reviewer:

Date:

REGION I
Data Review Worksheets

I. DATA COMPLETENESS

MISSING INFORMATION

DATE LAB CONTACTED

DATE RECD

REGION I
Data Review Worksheets

II. HOLDING TIMES

Complete table for all samples and circle the analysis date for samples not within criteria.

SAMPLE ID	DATE SAMPLED	HG DATE ANALYSIS	CYANIDE DATE ANALYSIS	OTHERS DATE ANALYSIS	pH	<u>ACTION</u>
----------------------	-------------------------	---------------------------------	--------------------------------------	-------------------------------------	-----------	----------------------

METALS - 180 DAYS FROM SAMPLE COLLECTION
MERCURY - 28 DAYS FROM SAMPLE COLLECTION
CYANIDE - 14 DAYS FROM SAMPLE COLLECTION

ACTION:

1. If holding times are exceeded all positive results are estimated (J) and non-detects are estimated (UJ).
2. If holding times are grossly exceeded, the reviewer may determine that non-detects are unusable (R).

REGION I
Data Review Worksheets

III A. INSTRUMENT CALIBRATION (Section 1)

1. Recovery Criteria

List the analytes which did not meet the percent recovery (%R) criteria for Initial or Continuing Calibration.

<u>DATE</u>	<u>ICV/CCV#</u>	<u>ANALYTE</u>	<u>%R</u>	<u>ACTION</u>	<u>SAMPLES AFFECTED</u>
-------------	-----------------	----------------	-----------	---------------	-------------------------

ACTIONS:

If any analyte does not meet the %R criteria follow the actions stated below:

For Positive Results:

	<u>Accept</u>	<u>Estimate (J)</u>	<u>Reject (R)</u>
Metals	90-110%R	75-89%R, 111-125%R	<75%R, >125%R
Mercury	80-120%R	65-79%R, 121-135%R	<65%R, >135%R
Cyanide	85-115%R	70-84%R, 116-130%R	<70%R, >130%R

For Non-detected Results:

	<u>Accept</u>	<u>Estimate (UJ)</u>	<u>Reject (R)</u>
Metals	90-125%R	75-89%R	<75%R, >125%R
Mercury	80-135%R	65-79%R	<65%R, >135%R
Cyanide	85-130%R	70-84%R	<70%R, >130%R

REGION I
Data Review Worksheets

III B. INSTRUMENT CALIBRATION (Section 2)

2. Analytical Sequence

- | | | |
|-----------|---|-----------|
| A. | Did the laboratory use the proper number of standards for calibration as described in the SW-846 (3rd Edition) manual. There is no difference on the proper number of standards between the CLP-SOW and SW-846. | Yes or No |
| B. | Were calibrations performed at the beginning of each day (or every 8 hours): whichever is more frequent. | Yes or No |
| C. | Were midpoint calibration standards analyzed at the beginning of sample analysis and at a minimum frequency of ten percent? | Yes or No |
| D. | Were the correlation coefficients for the calibration curves for AA, Hg, and CN ≤ 0.995 ? | Yes or No |

If No,

The data may be affected. Use professional judgement to determine the severity of the effect and qualify the data accordingly. Discuss any actions below and list the samples affected.

REGION I
Data Review Worksheet

IV A. BLANK ANALYSIS RESULTS (Sections 1-3)

List the blank contamination in Sections 1 & 2 below. A separate worksheet must be used for soil and water blanks.

MATRIX: _____

1. Laboratory Blanks

<u>DATE</u>	<u>ICB/CCB#</u>	<u>PREP BL</u>	<u>ANALYTE</u>	<u>CONC./UNITS</u>
-------------	-----------------	----------------	----------------	--------------------

2. Equipment/Trip Blanks

<u>DATE</u>	<u>EQUIP BL#</u>	<u>ANALYTE</u>	<u>CONC./UNITS</u>
-------------	------------------	----------------	--------------------

3. Frequency Requirements

- | | | |
|----|--|-----------|
| A. | Was a preparation blank carried through the entire analytical process for each matrix type or group of 20 samples, whichever is more frequent? | Yes or No |
| B. | Was a reagent blank with a minimum of three standards run daily? | Yes or No |
| C. | If 20 or more samples were run, was a reagent blank analyzed along with a mid-range standard at a frequency of every 10 samples? | Yes or No |

If No,

The data may be affected. Use professional judgement to determine the severity of the effect and qualify the data accordingly. Discuss any actions below, and list the samples affected.

REGION I
Data Review Worksheets

IV B. BLANK ANALYSIS RESULTS (Section 4)

4. Blank Actions

The Action Levels for any analyte is equal to five times the highest concentration of that element's contamination in any blank. The action level for samples which have been concentrated or diluted should be multiplied by the concentration/dilution factor. No positive sample result should be reported unless the concentration of the analyte in the sample exceeds the Action Level (AL). Specific actions are as follows:

1. When the concentration is greater than the IDL, but less than the Action Level, report the sample concentration detected with a U.
2. When the sample concentration is greater than the Action Level, report the sample concentration unqualified.

MATRIX: _____

MATRIX: _____

<u>ELEMENT</u>	<u>MAX. CONC./</u> <u>UNITS</u>	<u>AL/</u> <u>UNITS</u>
----------------	------------------------------------	----------------------------

<u>ELEMENT</u>	<u>MAX. CONC./</u> <u>UNITS</u>	<u>AL/</u> <u>UNITS</u>
----------------	------------------------------------	----------------------------

NOTE: Blanks analyzed during a soil case must be converted to mg/kg in order to compare them with the sample results.

$$\text{conc. in ug/l} \times \frac{\text{volume diluted to (100 ml)}}{\text{weight digested (1 gram)}} \times \frac{1 \text{ liter}}{1000 \text{ ml}} \times \frac{1000 \text{ gms.}}{1 \text{ kg}} \times \frac{1 \text{ mg}}{1000 \text{ ug}} = \frac{\text{mg}}{\text{kg}}$$

Multiplying this result by 5 to arrive at the action level gives a final result in mg/kg which can then be compared to sample results.

The SW-846 (3rd Edition) requirement is that the calibration blank be within a three (3) standard deviation window of the mean blank value. As such, gross blank contamination warrants the data validator to contact the laboratory to verify this was performed. List all anomalies in the Inorganic Regional Data Assessment.

REGION I
Data Review Worksheets

V A. ICP INTERFERENCE CHECK SAMPLE (Sections 1 & 2)

1. Recovery Criteria

List any elements in the ICS AB solution which did not meet the criteria for %R. SW-846 (3rd Edition) does warrant a $\pm 20\%$ window of the true value. The laboratory in accordance with SW-846 Method 6010 must follow an established control limit of 1.5 times the standard deviation of the mean value. If reoccurring problems arise, contact the lab and determine if any deviation from this procedure has occurred.

<u>DATE</u>	<u>ELEMENT</u>	<u>%R</u>	<u>ACTION</u>	<u>SAMPLES AFFECTED</u>
-------------	----------------	-----------	---------------	-------------------------

ACTIONS:

If an element does not meet the %R criteria, follow the actions stated below:

	<u>PERCENT RECOVERY</u>		
	<50%	50-79%	>120%
Positive Sample Results	R	J	J
Non-detected Sample Results	R	UJ	A

2. Frequency Requirements

Were Interference QC samples run at the beginning and end of each batch analysis run or a minimum of twice per 8 hour working shift, whichever is more frequent?

Yes or No

If no,

The data may be affected. Use professional judgement to determine the severity of the effect and qualify the data accordingly. Discuss any actions below and list the samples affected.

REGION I
Data Review Worksheets

V B. ICP INTERFERENCE CHECK SAMPLE (Section 3)

3. Report the concentration of any elements detected in the ICS A solution > 2xIDL that should not be present.

<u>ELEMENT</u>	<u>CONC. DETECTED IN THE ICS</u>	<u>CONC. OF INTERFERENTS IN THE ICS</u>			
		<u>AL</u>	<u>CA</u>	<u>FE</u>	<u>MG</u>

Estimate the concentration produced by the interfering element in all affected samples. See guidelines for examples. List the samples affected by interferences below:

<u>SAMPLE AFFECTED</u>	<u>ELEMENT AFFECTED</u>	<u>SAMPLE CONC. (ug/L)</u>	<u>SAMPLE INTERFERENT CONC.</u>				<u>ESTIMATED INTERF. (ug/L)</u>
			<u>AL</u>	<u>CA</u>	<u>FE</u>	<u>MG</u>	

ACTIONS:

1. In general, the sample data can be accepted without qualification if the sample concentrations of Al, Ca, Fe, and Mg are less than 50% of their respective levels in the ICS solution.
2. Estimate (J) positive results for affected elements for samples with levels of interferents 50% or more of that in the ICS solution.
3. Reject (R) positive results if the reported concentration is due entirely to the interfering element.
4. Estimate (UJ) non-detected results for which false negatives are suspect.

Give explanations for any actions taken below:

REGION I
Data Review Worksheets

VI. MATRIX SPIKE

TR # _____

MATRIX: _____

1. Recovery Criteria

List the percent recoveries for analytes which did not meet the required criteria.

S - amount of spike added
SSR - spikes sample request
SR - sample result

Analyte	SSR	SR	S	%R	Action
---------	-----	----	---	----	--------

Matrix Spike Actions apply to all samples of the same matrix.

ACTIONS:

- If the sample concentration exceeds the spike concentration by a factor of 4 or more, no action is taken.
- If any analyte does not meet the %R criteria follow the actions stated below:

	<u>PERCENT RECOVERY</u>		
	<u><30%</u>	<u>30%-74%</u>	<u>>125%</u>
Positive Sample Results	J	J	J
Non-detected Results	R	UJ	No Action

2. Frequency Criteria

- Was a matrix spike prepared at the required frequency?
Yes or No

A separate worksheet should be used for each matrix spike pair.

VII. LABORATORY DUPLICATES

List the concentrations of any analyte not meeting the criteria for duplicate precision. For soil duplicates, calculate the PQL in mg/kg using the sample weight, volume and percent solids data for the sample. Indicate what criteria was used to evaluate precision by circling either the RPD or PQL for each element.

MATRIX: _____

<u>Element</u>	<u>PQL*</u>		<u>Sample #</u>	<u>Duplicate #</u>	<u>RPD**</u>	<u>Action</u>
	<u>water</u> ug/L	<u>soil</u> mg/kg				
Antimony	30					
Arsenic	2					
Barium	10					
Beryllium	2					
Cadmium	5					
Chromium	10					
Cobalt	20					
Copper	20					
Lead	2					
Mercury	0.2					
Nickel	20					
Selenium	2					
Silver	10					
Thallium	2					
Tin	100					
Vanadium	20					
Zinc	20					
Cyanide	10					

Laboratory Duplicate Actions should be applied to all other samples of the same matrix type.

ACTIONS:

1. Estimate (J) positive results for elements which have an RPD >20% for waters and >35% for soils.
 2. If sample results are less than 5x the PQL, estimate (J) positive results for elements whose absolute difference is >PQL, (2xPQL for soils). If both samples are non-detected, the RPD is not calculated (NC).
- * No specific detection limits are required in SW-846. All PQLs are of a recommended laboratory calculated nature. As such, all CLP-CRDLs will be substituted with the Radian calculated PQLs for reporting purposes.
- ** Mean RPD of $\pm 20\%$ or ± 2 standard deviations of the last 25 runs (whichever is tighter) for all analytes as warranted by SW-846 (3rd Edition) protocols will be utilized.

VIII. FIELD DUPLICATES

List the concentrations of all analytes in the field duplicate pair. For soil duplicates, calculate the PQL in mg/kg using the sample weight, volume and percent solids data for the sample. Indicate what criteria was used to evaluate the precision by circling either the RPD or PQL for each element.

MATRIX: _____

<u>Element</u>	<u>PQL</u>		<u>Sample #</u>	<u>Duplicate #</u>	<u>RPD**</u>	<u>Action</u>
	<u>water</u> ug/L	<u>soil</u> mg/kg				
Antimony	30					
Arsenic	2					
Barium	10					
Beryllium	2					
Cadmium	5					
Chromium	10					
Cobalt	20					
Copper	20					
Lead	2					
Mercury	0.2					
Nickel	20					
Selenium	2					
Silver	10					
Sodium	5000					
Thallium	2					
Tin	100					
Vanadium	20					
Zinc	20					
Cyanide	10					

Field Duplicate actions should be applied to all other samples of the same matrix type.

ACTIONS:

1. Estimate (J) positive results for elements which have an RPD >30% for waters and >50% for soils.
 2. If sample results are less than 5x the PQL, estimate (J) positive results and (UJ) nondetected results for elements whose absolute difference is >2xPQL, (4xPQL for soils). If both samples are non-detected, the RPD is not calculated (NC).
- No specific detection limits are required in SW-846. All PQLs are of a recommended laboratory calculated nature. As such, all CLP-CRDLs will be substituted with the Radian calculated PQLs for reporting purposes.
 - ** Mean RPD of $\pm 20\%$ or ± 2 standard deviations of the last 25 runs (whichever is tighter) for all analytes as warranted based on SW-846 (3rd Edition) protocols will be utilized.

IX. LABORATORY CONTROL SAMPLE

1. Aqueous LCS

List any LCS recoveries not within the 80-120% criteria and the samples affected.

<u>DATE</u>	<u>ELEMENT</u>	<u>%R</u>	<u>ACTION</u>	<u>SAMPLES AFFECTED</u>
-------------	----------------	-----------	---------------	-------------------------

Note: The SW-846 (3rd Edition) LCS recovery window is $\pm 20\%$. The current 80 - 100% window is acceptable.

2. Solid LCS

List any analytes that were not within the control windows set by the EPA manufacturers for the solid LCS sample. Lot specifications are available on request from Radian.

<u>ELEMENT</u>	<u>LCS CONC.</u>	<u>CONTROL WINDOWS</u>	<u>ACTION</u>	<u>SAMPLES AFFECTED</u>
----------------	------------------	------------------------	---------------	-------------------------

ACTIONS:

<u>AQUEOUS LCS</u>	<u><50%</u>	<u>51-79%</u>	<u>>120%</u>
--------------------	----------------	---------------	-----------------

Positive Results	R	J J	
Non-detected Results	R	UJ A	

<u>SOLID LCS</u>	<u><EPA Control Windows</u>	<u>>EPA Control Windows</u>
------------------	--------------------------------	--------------------------------

Positive Results	J J
Non-detected Results	UJ A

3. Frequency Criteria

A. Was an LCS analyzed for every matrix, every digestion batch, and every 20 samples?	Yes or No
---	-----------

CIBA-GEIGY
ORGANIC REGION I WORKSHEETS
RE-EDITED FOR APPENDIX IX
CONSTITUENTS

Prepared by:

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CIBA-GEIGY
ORGANIC REGION I WORKSHEET

Background: The term hazardous constituent used in the Solid Waste Disposal Act Section 3004(u) means constituents found in Appendix VIII to 40CFR part 261. EPA also defines those constituents identified in Appendix IX to 40CFR part 264. Appendix IX constituents generally constitutes a subset of Appendix VIII particularly suitable for ground water analyses.

In general, where very little is known of waste characteristics, and where there is a potential for a wide spectrum of wastes to have been released, only then is the owner/operator required to perform an extensive routine analysis for a broader spectrum of waste such as an Appendix IX analysis.

Radian Corporation of Austin, Texas, has been sub-contracted by WCC to analyze the 218 organic hazardous constituents in Appendix IX and will be utilizing the following SW-846 procedures listed in Table 1. As such, the enclosed Organic Region I Data Validation Worksheets have been modified accordingly for each fraction to conform to the QA/QC criteria of each SW-846 test methods in Table 1.

TABLE 1
SELECTED ANALYTICAL METHODS FOR
ORGANIC APPENDIX IX ANALYSES

SW-846 Method	General Category/ Analyte	Technique	Number of Analytes Measured
8080	Organochlorine Pesticides and PCBs	GC/ECD	28
8140	Organophosphorus Pesticides	GC/FPD	9
8150	Herbicides	GC/ECD	4
8240	Volatile Organics	GC/MS	*54
8270	Semivolatile Organics	GC/MS	**111
8280	Dioxins and Furans	GC/MS	12
		TOTAL	218

GC/ECD - Gas Chromatography/Electron Capture Detection
GC/EPD - Gas Chromatography/Flame Photometric Detection
GC/MS - Gas Chromatography/Mass Spectrometry

- * This number includes three analytes (1,4-Dioxane, isobutanol, methacrylonitrile) that will be analyzed by Method 8240 Direct Injection.
- ** This number includes Appendix IX analytes, however, this number will increase based on site specific compounds that will be analyzed by Method 8270, also. Sym-Trinitrobenzene will be analyzed as a tentatively identified compound due to unavailability of standard.

ORGANIC
SUMMARY TABLES

DATA SUMMARY FORM: VOLATILES 1

Appendix IX
WATER SAMPLES
(µg/L)

Site Name: _____

Case #: _____ Sampling Date(s): _____

To calculate sample quantitation limit:
(CRL * Dilution Factor)
PQL

Sample No. Dilution Factor Location																			
PQL CRL	COMPOUND	Conc.	Q	Conc.	Q	Conc.	Q	Conc.	Q	Conc.	Q	Conc.	Q	Conc.	Q	Conc.	Q	Conc.	Q
10	Chloromethane																		
10	Bromomethane																		
10	*Vinyl Chloride																		
10	Chloroethane																		
105	*Methylene Chloride																		
100	Acetone																		
5	Carbon Disulfide																		
5	*1,1-Dichloroethene																		
5	1,1-Dichloroethane																		
5	*Total 1,2-Dichloroethene																		
5	Chloroform																		
5	*1,2-Dichloroethane																		
100	*2-Butanone (MEK)																		
5	*1,1,1-Trichloroethane																		
5	*Carbon Tetrachloride																		
10	Vinyl Acetate																		
5	Bromodichloromethane																		
75	ACROLEIN																		
50	ACRYLONITRILE																		
20	ACETONITRILE																		
5	3-CHLOROPROPENE																		
25	2-CHLORO-1,3-BUTADIENE																		
10,000	1,4-DIOXANE																		
20	DICHLORODIFLUOROMETHANE																		

QL = Contract Required Quantitation Limit

Action Level Exists

SEE NARRATIVE FOR CODE DEFINITIONS

PQL = Practical Quantitation Limit

revised 07/90

Q = data validation quality

10

Prepared by: JPL (Woodward-Clude)

DATA SUMMARY FORM: VOLATILES 2

Appendix IX
WATER SAMPLES
(µg/L)

Site Name: _____

Case #: _____ Sampling Date(s): _____

To calculate sample quantitation limit:
(CRQL * Dilution Factor)
PRL

Sample No. Dilution Factor Location																	
PQL CRQL	COMPOUND	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q
5	*1,2-Dichloropropane																
5	Cis-1,3-Dichloropropane																
5	Trichloroethene																
5	Dibromochloromethane																
5	1,1,2-Trichloroethane																
5	*Benzene																
5	Trans-1,3-Dichloropropene																
5	Bromoform																
50,10	4-Methyl-2-pentanone																
50,10	2-Hexanone																
5	*Tetrachloroethene																
5	1,1,2,2-Tetrachloroethane																
5	*Toluene																
5	*Chlorobenzene																
5	*Ethylbenzene																
5	*Styrene																
5	*Total Xylenes																
5	TRANS-1,2-DICHLOROETHENE																
10	1,2-DIBROMOETHANE																
10	TRANS-1,4-DICHLORO-2-BUTENE																
20	1,2-DIBROMO-3-CHLOROPROPANE																
10	ETHYL METHACRYLATE																
5	ISOMETHANE																
10,000	ISOBUTANOL																

CRQL = Contract Required Quantitation Limit

*Action Level Exists

SEE NARRATIVE FOR CODE DEFINITIONS

PQL = Practical Quantitation Limit

revised 07/90

Q = Data Validation Qualifier

10

Prepared by: JPL (Woodward-Clark)

DATA SUMMARY FORM: VOLATILES 1

Appendix II

SOIL SAMPLES

(µg/Kg)

Site Name: _____

Use #: _____ Sampling Date(s): _____

To calculate sample quantitation limit:

$$\frac{(\text{CRL} * \text{Dilution Factor})}{((100 - \% \text{ moisture})/100)}$$
 PAL

PAL CRL	COMPOUND	Sample No. Dilution Factor % Moisture Location															
		conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q
2000	Chloromethane																
2000	Bromomethane																
2000	Vinyl Chloride																
2000	Chloroethane																
2000	Methylene Chloride																
2000	Acetone																
1000	Carbon Disulfide																
1000	1,1-Dichloroethene																
1000	1,1-Dichloroethane																
1000	1,2-Dichloroethene																
1000	Chloroform																
1000	1,2-Dichloroethane																
2000	2-Butanone (MEK)																
1000	1,1,1-Trichloroethane																
1000	Carbon Tetrachloride																
1000	Vinyl Acetate																
1000	Bromodichloromethane																
1500	ACROLEIN																
1000	ACRYLONITRILE																
400	ACETONITRILE																
100	3-CHLOROPROPENE																
500	2-CHLORO-1,3-BUTADIENE																
10,000	1,4-DIOXANE																
400	DICHLOROFLUOROMETHANE																

CRL = Contract Required Quantitation Limit

PAL = Practical Quantitation Limit (wet-weight basis): samples results on a dry weight basis will yield higher PAL (refer to calculation: top right)

SEE NARRATIVE FOR CODE DEFINITIONS

revised 07/90

10

Q = data validation qualities

Prepared by: JPL/Woodward, P.D.

DATA SUMMARY FORM: VOLATILES 2

Appendix IX

SOIL SAMPLES

(µg/Kg)

Site Name: _____

Case #: _____ Sampling Date(s): _____

To calculate sample quantitation limit:
 $(CRL \times \text{Dilution Factor}) / ((100 - \% \text{ moisture})/100)$
 PQL

PQL CRL	COMPOUND	Sample No. Dilution Factor % Moisture Location															
		conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q
100.5	1,2-Dichloropropane																
100.5	Cis-1,3-Dichloropropene																
100.5	Trichloroethene																
100.5	Dibromochloromethane																
100.5	1,1,2-Trichloroethane																
100.5	Benzene																
100.5	Trans-1,3-Dichloropropene																
100.5	Bromoform (TRIBROMOMETHANE)																
100.5	4-Methyl-2-pentanone																
100.5	2-Hexanone																
100.5	Tetrachloroethene																
100.5	1,1,2,2-Tetrachloroethane																
100.5	Toluene																
100.5	Chlorobenzene																
100.5	Ethylbenzene																
100.5	Styrene																
100.5	Total Xylenes																
100	TRANS-1,2-DICHLOROETHENE																
200	1,2-DIBROMOETHANE																
200	TRANS-1,4-DICHLORO-2-BUTENE																
100	1,2-DIBROMO-3-CHLOROPROPANE																
200	ETHYL METHACRYLATE																
100	IDONETHANE																
10,000	ISOBUTANOL																

CRL = Contract Required Quantitation Limit

SEE NARRATIVE FOR CODE DEFINITIONS

PQL = Practical Quantitation Limit (wet-weight basis); sample results on
 a dry weight basis will yield a higher PQL (refer to calculation: top right)

Q = Data Validation Qualities revised DT/90
 10

Site Name: _____

Case #: **Sampling Date(s):**

To calculate sample quantitation limits:
(~~CEQL~~ * Dilution Factor)
PQL

[illegible]

Action Level Exists

Q = Data Validation Quality

Prepared by: John Lorenzo (wcc)¹⁰

DATA SUMMARY FORM: B N A S 2

Site Name: _____

WATER SAMPLES

(µg/L)

Case #: _____ Sampling Date(s): _____

To calculate sample quantitation limit:

(CRL * Dilution Factor)

PQL

Sample No. Dilution Factor Location																			
PQL CRL	COMPOUND	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q
10	Hexachlorobutadiene																		
10	4-Chloro-3-methylphenol																		
10	2-Methylnaphthalene																		
10	Hexachlorocyclopentadiene																		
10	2,4,6-Trichlorophenol																		
50	2,4,5-Trichlorophenol																		
10	2-Chloronaphthalene																		
50	2-Nitroaniline																		
10	Dimethylphthalate																		
10	Acenaphthylene																		
10	2,6-Dinitrotoluene																		
50	3-Nitroaniline																		
10	Acenaphthene																		
50	2,4-Dinitrophenol																		
50	4-Nitrophenol																		
10	Dibenzofuran																		
10	2,4-Dinitrotoluene																		
10	Diethylphthalate																		
10	4-Chlorophenyl-phenylether																		
10	Fluorene																		
50	4-Nitroaniline																		
50	4,6-Dinitro-2-methylphenol																		
50	1-AMINOBI-PHENYL																		
50	2-ACETYLAMINOFLUORENE																		

CRL = Contract Required Quantitation Limit

PQL

PRACTICAL

Q = Data Validation Qualifier

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Prepared by: John Lorenzo (wcc)

DATA SUMMARY FORM: B N A S 3

Site Name: _____

WATER SAMPLES
(µg/L)

Case #: _____ Sampling Date(s): _____

To calculate sample quantitation limit:
 (CRQL * Dilution Factor)
 PQL

Sample No. Dilution Factor Location																	
PQL CRQL	COMPOUND	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q
10	N-Nitrosodiphenylamine																
10	4-Bromophenyl-phenylether																
10	*Hexachlorobenzene																
50	*Pentachlorophenol																
10	Phenanthrene																
10	Anthracene																
10	Di-n-butylphthalate																
10	Fluoranthene																
10	Pyrene																
10	Butylbenzylphthalate																
20	3,3'-Dichlorobenzidine																
10	Benzo(a)anthracene																
10	Chrysene																
10	bis(2-Ethylhexyl)phthalate																
10	Di-n-octylphthalate																
10	Benzo(b)fluoranthene																
10	Benzo(k)fluoranthene																
10	Benzo(a)pyrene																
10	Indeno(1,2,3-cd)pyrene																
10	Dibenz(a,h)anthracene																
10	Benzo(g,h,i)perylene																
100	ARAMITE																
10	CHLORO BENZILATE																
10	DIMETHYLPHENETHYLAMINE																

CRQL = Contract Required Quantitation Limit

PQL Potential

Q = Data Validation Qualifier

Action Level Exists

SEE NARRATIVE FOR CODE DEFINITIONS

revised 07/90

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Prepared by: John Lorenzo (wcc)

BNA
DATA SUMMARY FORM: ORGANICS 4

Site Name: _____

WATER SAMPLES
(µg/L)

Case #: _____ Sampling Date(s): _____

To calculate sample quantitation limit:
(QL * Dilution Factor)
p

Sample No. Dilution Factor Location																			
PQL	Compound	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q
10	2,6-DICHLOROPHENOL																		
10	1,3-DINITROBENZENE																		
10	DIPHENYLAMINE																		
30	P-DIMETHYLAMINOAZOBENZENE																		
20	3,3'-DIMETHYLBENZIDINE																		
50	7,12-DIMETHYLBENZ(A)ANTHRACENE																		
10	DIALATE																		
10	ETHYL METHANESULFONATE																		
50	HEXACHLOROPROPENE																		
50	HEXACHLOROPHENE																		
10	ISOSAFROLE																		
50	METHYL METHANESULFONATE																		
10	3-METHYLPHENOL(M-CRESOL)																		
40	METHAPYRILINE																		
30	3-METHYLCHOLANTHRENE																		
10	N-NITROSODIMETHYLAMINE																		
10	N-NITROSONETHYLETHYLAMINE																		
10	N-NITROSDIETHYLAMINE																		
10	N-NITROSPYRROLIDINE																		
10	N-NITROSMORPHOLINE																		
10	N-NITROPIPERIDINE																		
20	N-NITROSDI-N-BUTYLAMINE																		
10	1,4-NAPTHOQUINONE																		
20	1-NAPHTHYLAMINE																		

QL = Quantitation Limit

Practical

Q = Data Validation Qualifier

SEE NARRATIVE FOR CODE DEFINITIONS

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BNA
DATA SUMMARY FORM: ORGANICS 5

WATER SAMPLES
(µg/L)

To calculate sample quantitation limit:
(QL = Dilution Factor)
P

Site Name: _____
Use #: _____ Sampling Date(s): _____

PQL	Compound	Sample Number		Dilution Factor		Location													
		conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q
660	2-NAPHTHYLAMINE																		
660	5-NITRO-O-TOLUIDINE																		
3000	4-NITROQUINOLINE-N-OXIDE																		
660	PIRIDINE																		
2300	2-PICOLINE																		
330	PENTACHLOROETHANE																		
660	PENTACHLOROBENZENE																		
330	PHENACETIN																		
660	PENTACHLORONITROBENZENE																		
1000	PROPAHIDE																		
1700	P-PHENYLENE DIANINE																		
330	SAFROLE																		
330	O-TOLUIDINE																		
330	1,2,4,5-TETRACHLOROBENZENE																		
660	2,3,4,6-TETRACHLOROPHENOL																		
	TINUVIN 327																		
	IRGASAN DP-300																		
	PROPAZINE																		
	BUTAZOLIDEN																		
	TOFRANIL																		
	DCDD																		
	TRCDD																		
	DCDF																		
	TRCDF																		

QQL = Quantitation Limit

NOTE:

SYN-TRINITROBENZENE WAS ANALYZED AS A TENTATIVELY IDENTIFIED COMPOUND DUE TO THE UNAVAILABILITY OF STANDARDS

SEE NARRATIVE FOR CODE DEFINITIONS

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DATA SUMMARY FORM: B N A S 1

Site Name: _____

SOIL SAMPLES
(µg/Kg)

Case #: _____ Sampling Date: _____

To calculate sample quantitation limit:
 $(\text{CRQL} * \text{Dilution Factor}) / ((1 - \% \text{ moisture})/100)$
 PQL

PQL CRQL	COMPOUND	Sample No. Dilution Factor % Moisture Location															
		conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q
330	Phenol																
330	bis(2-Chloroethyl)ether																
330	2-Chlorophenol																
330	1,3-Dichlorobenzene																
330	1,4-Dichlorobenzene																
330	Benzyl Alcohol																
330	1,2-Dichlorobenzene																
330	2-Methylphenol (<i>o</i> -cresol)																
330	bis(2-Chloroisopropyl)ether																
330	4-Methylphenol																
330	N-Nitroso-di-n-propylamine																
330	Hexachloroethane																
330	Nitrobenzene																
330	Isophorone																
330	2-Nitrophenol																
330	2,4-Dimethylphenol																
4600	Benzoic Acid																
330	bis(2-Chloroethoxy)methane																
330	2,4-Dichlorophenol																
330	1,2,4-Trichlorobenzene																
330	Naphthalene																
330	4-Chloroaniline																
330	ACETOPHENONE																
1700	ANILINE																

CRQL = Contract Required Quantitation Limit
 PQL Practical

SEE NARRATIVE FOR CODE DEFINITIONS
 revised 07/90

Prepared by: John Loung (wcc)

DATA SUMMARY FORM: B N A S 2

Site Name: _____

SOIL SAMPLES

(µg/Kg)

Case #: _____ Sampling Date(s): _____

To calculate sample quantitation limit:
 $(CRL \times \text{Dilution Factor}) / ((100 - \% \text{ moisture})/100)$
 PQL

PQL CRL	COMPOUND	Sample No. Dilution Factor % Moisture Location															
		conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q
330	Hexachlorobutadiene																
330	4-Chloro-3-methylphenol																
330	2-Methylnaphthalene																
330	Hexachlorocyclopentadiene																
330	2,4,6-Trichlorophenol																
1600 ⁷	2,4,5-Trichlorophenol																
330	2-Chloronaphthalene																
1600	2-Nitroaniline																
330	Dimethylphthalate																
330	Acenaphthylene																
330	2,6-Dinitrotoluene																
1600 ⁷	3-Nitroaniline																
330	Acenaphthene																
1600 ⁷	2,4-Dinitrophenol																
1600 ⁷	4-Nitrophenol																
330	Dibenzofuran																
330	2,4-Dinitrotoluene																
330	Diethylphthalate																
330	4-Chlorophenyl-phenylether																
330	Fluorene																
1600 ⁷	4-Nitroaniline																
1600 ⁷	4,6-Dinitro-2methylphenol																
1700	4-AMINOBIPHENYL																
1700	2-ACETYLAMINOFLORENE																

CRL = Contract Required Quantitation Limit

PQL = Practical Quantitation Limit

Q = Data Validation Qualifier

SEE NARRATIVE FOR CODE DEFINITIONS

revised 07/90

10

Prepared by: John Lorenzo (WCC)

DATA SUMMARY FORM: B N A S. 3

Site Name: _____

SOIL SAMPLES
(µg/Kg)

Case #: _____ Sampling Date(s): _____

To calculate sample quantitation limit:
 $(\text{CRQL} * \text{Dilution Factor}) / ((100 - \% \text{ moisture})/100)$
 PQL

PQL CRQL	COMPOUND	Sample No. Dilution Factor % Moisture Location				Conc. Q		Conc. Q		Conc. Q		Conc. Q		Conc. Q		Conc. Q		Conc. Q	
330	N-Nitrosodiphenylamine																		
330	4-Bromophenyl-phenylether																		
330	Hexachlorobenzene																		
1600	Pentachlorophenol																		
330	Phenanthrene																		
330	Anthracene																		
330	Di-n-butylphthalate																		
330	Fluoranthene																		
330	Pyrene																		
330	Butylbenzylphthalate																		
660	3,3'-Dichlorobenzidine																		
330	Benzo(a)anthracene																		
330	Chrysene																		
330	bis(2-Ethylhexyl)phthalate																		
330	Di-n-octylphthalate																		
330	Benzo(b)fluoranthene																		
330	Benzo(k)fluoranthene																		
330	Benzo(a)pyrene																		
330	Indeno(1,2,3-cd)pyrene																		
330	Dibenz(a,h)anthracene																		
330	Benzo(g,h)perylene																		
3000	ARAMITE																		
330	CHLOROBENZILATE																		
330	DINETHYLPHENETHYLAMINE																		

CRQL = Contract Required Quantitation Limit

PQL = Practical Quantitation Limit

Q = Data Validation Qualifier

SEE NARRATIVE FOR CODE DEFINITIONS

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Prepared by: John Lorenzo (wcc)

BNA
DATA SUMMARY FORM: ORGANICS 4

Site Name: _____

SOIL SAMPLES
(µg/Kg)

Case #: _____ Sampling Date(s): _____

To calculate sample quantitation limit:
 $(QL * \text{Dilution Factor}) / ((100 - \% \text{ moisture})/100)$
P

Sample Number Dilution Factor Location % moisture																	
PQL	Compound	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q
330	2,6-DICHLOROPHENOL																
330	1,3-DINITROBENZENE																
330	DIPHENYLAMINE																
1000	P-DIMETHYLANNOAZOBENZENE																
2100	3,3'-DIMETHYLBENZIDINE																
1700	7,12-DIMETHYLBENZ(A)ANTHRACENE																
330	DIALATE																
330	ETHYL METHANESULFONATE																
1700	HEXACHLOROPROPENE																
1700	HEXACHLOROPHENE																
330	ISOSAFROLE																
1700	METHYL METHANESULFONATE																
330	3-METHYLPHENOL (M-CRESOL)																
1300	METHAPYRILNE																
1000	3-METHYLCHOLANTHRENE																
330	N-NITROSODIMETHYLAMINE																
330	N-NITROSOMETHYLETHYLAMINE																
330	N-NITROSODIETHYLAMINE																
330	N-NITROSOPYRROLIDINE																
330	N-NITROSOMORPHOLINE																
330	N-NITROPIPERIDINE																
660	N-NITROSO-DE-N-BUTYLAMINE																
330	1,4-NAPHOQUINONE																
660	1-NAPHTHYLAMINE																

PQL = Quantitation Limit
Practical
Q = Data Validation Qualifier

SEE NARRATIVE FOR CODE DEFINITION
revised 07/9
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BNA
DATA SUMMARY FORM: ORGANICS 5

SOIL SAMPLES
(µg/Kg)

Site Name: _____

Case #: _____ Sampling Date(s): _____

To calculate sample quantitation limit:
(QL = Dilution Factor) / ((100 - % moisture)/100)

Sample Number Dilution Factor Location % Moisture															
PQL	Compound	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q
20	2-NAPHTHYLAMINE														
20	5-NITRO-O-TOLUIDINE														
100	4-NITROQUINOLINE-N-OXIDE														
20	PYRIDINE														
70	2-PICOLINE														
20	PENTACHLOROETHANE														
20	PENTACHLOROBENZENE														
10	PHENACETIN														
20	PENTACHLORONITROBENZENE														
30	PRONAMIDE														
50	P-PHENYLENEDIAMINE														
10	SAFROLE														
10	O-TOLUIDINE														
10	1,2,4,5-TETRACHLOROBENZENE														
20	2,3,4,6-TETRACHLOROPHENOL														
	TINUVIN 329														
	IRGASAN DP-300														
	PROPAZINE														
	BUTAZOLIDIN														
	TOFRANIL														
	DCDD														
	TRCDD														
	DCOF														
	TRCOF														

SYN-TRINITROBENZENE
QL = Quantitation Limit

SEE NARRATIVE FOR CODE DEFINITIONS

revised 01/90

PQL PRACTICAL NOTE: SYN-TRINITROBENZENE WAS ANALYZED AS A TENTATIVELY IDENTIFIED COMPOUND DUE TO THE

UNAVAILABILITY OF STANDARDS

Q = Data Validation Qualifier

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DATA SUMMARY FORM: PESTICIDES AND PCB'S 1
DIOXINS / CLASSICAL PARAMETERS
WATER SAMPLES
(µg/L)

Site Name: _____

Case #: _____ Sampling Date(s): _____

To calculate sample quantitation limit:
 $(CRL \div \text{Dilution Factor})$
PQL

Sample No. Dilution Factor Location																	
PQL CRL	COMPOUND	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q
0.05	alpha-BHC																
0.05	beta-BHC																
0.05	delta-BHC																
0.05	*gamma-BHC (Lindane)																
0.05	*Heptachlor																
0.05	Aldrin																
0.05	Heptachlor Epoxide																
0.05	Endosulfan I																
0.10	Dieldrin																
0.10	4,4'-DDE																
0.10	*Endrin																
0.10	Endosulfan II																
0.10	4,4'-DDD																
0.10	Endosulfan Sulfate																
0.10	4,4'-DDT																
0.50	*Methoxychlor																
0.10	Endrin Ketone																
0.50	*alpha-Chlordane																
0.50	*gamma-Chlordane																
1.0	*Toxaphene																
0.50	*Aroclor-1016																
0.50	*Aroclor-1221																
0.50	*Aroclor-1232																
0.50	*Aroclor-1242																
0.50	*Aroclor-1248																
1.0	*Aroclor-1254																
1.0	*Aroclor-1260																

CRL = Contract Required Quantitation Limit
PQL = Practical

Q = Data Validation Qualifier

Action Level Exists

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DATA SUMMARY FORM: O R G A N I C S 2

WATER SAMPLES
($\mu\text{g/L}$)

Site Name: _____

Case #: _____ Sampling Date(s): _____

Reference

**To calculate sample quantitation limit:
(QL * Dilution Factor)**

[illegible]

PQL = Quantitation Limit

Piacental

Q = Data Validation Qualifier

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SOIL SAMPLES
(µg/Kg)

To calculate sample quantitation limit:
 (QL * Dilution Factor) / ((100 - % moisture)/100)

Reference

PQL = Quantitation Limit
Practical
Q = Data Validated Qualities

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CIBA-GEIGY, Cranston, Rhode Island
REGION I
Data Review Worksheets
Re-Edited for Appendix IX
Constituent Analysis

Reference No.:

REGION I REVIEW OF ORGANIC CONTRACT LABORATORY DATA PACKAGE

The hardcopied (laboratory name) _____ data package received at Region I has been reviewed and the quality assurance and performance data summarized. The data review included:

Matrix:
No. of Samples:
Trip Blank No.:
Equipment Blank No.:
Field Dup Nos.:
Sampling Date(s):
Shipping Date(s):
Date Rec'd by Lab:
Sample Identifiers:

SW-846 (3rd Edition) requires that specific analytical work be done. The general criteria used to determine the performance were based upon an examination of:

- | | |
|-----------------------|--------------------------------|
| -Data Completeness | -Matrix Spike/Matrix Spike Dup |
| -Holding Times | -Field Duplicates |
| -GC/MS Tuning | -Internal Standard Performance |
| -Calibrations | -Pesticide Inst. Performance |
| -Blanks | -Compound Identification |
| -Surrogate Recoveries | -Compound Quantification |

Overall Comments:

Definitions and Qualifiers:

- A - Acceptable data.
- J - Approximate data due to quality control criteria.
- R - Reject data due to quality control criteria
- U - Analyte not detected

Reviewer:

Date:

REGION I
Data Review Worksheets

I. DATA COMPLETENESS

MISSING INFORMATION

DATE LAB CONTACTED

DATE REC'D

REGION I
Data Review Worksheets

II. HOLDING TIMES

Complete table for all samples and circle the fractions which are not within criteria.

SAMPLE ID	DATE SAMPLED	VOA	BNA		PEST	
		DATE ANAL	DATE EXTR	DATE ANAL	DATE EXTR	DATE ANAL

In accordance with Table 4-1 of Section 4.0 SW-846 and EPA Region I protocols the holding time criteria and action levels are:

VOA - Unpreserved: Aromatic within 7 days, non-aromatic within 14 days of sample collection.

Preserved: Both within 14 days of sample collection.

Soils: Both within 14 days of sample collection.

BNA & PEST - Water: Extracted within 7 days, analyzed within 40 days.
Soils (Solids): Extracted within 14 days, analyzed within 40 days.

- ACTION:
1. If holding times are exceeded, all positive results are estimate (J) and non-detects are estimated (UJ).
 2. If holding times are grossly exceeded, the reviewer may determine that non-detects are also unusable.

REGION I
Data Review Worksheets

III. GC/MS TUNING

The DFTPP performance results were reviewed and found to be with the specified criteria.

If no,
Samples affected:

The BFB performance results were reviewed and found to be within the specified criteria.

If no,
Samples affected:

If mass calibration is in error refer to the Region guidelines for expanded criteria. If necessary, qualify all associated data as unusable (R).

Note: The data reviewer must realize that the BFB and DFTPP tune criteria for CLP and SW846 protocols are the same.

REGION I
Data Review Worksheet

IV A. VOLATILE CALIBRATION VERIFICATION

Date of Initial Calibration :
Dates of Continuing Calibrations:
Instrument ID :
Matrix/Level :

<u>DATE</u>	<u>CRITERIA OUT</u> RF,%RSD,RF,%D	<u>COMPOUND (VALUE)</u>
_____	Samples Affected:	
_____	Samples Affected:	
_____	Samples Affected:	
_____	Samples Affected:	
_____	Samples Affected:	
_____	Samples Affected:	
_____	Samples Affected:	
_____	Samples Affected:	
_____	Samples Affected:	
_____	Samples Affected:	

1. All RF's must be >0.05
2. All %RSD's must be $<30\%$
3. All %D's must be $<25\%$

ACTION:

1. If any compound has an initial RF or a continuing RF of <0.05 :
 - a. Flag positive results for that compound as estimated (J).
 - b. Flag non-detects for that compound as unusable (R).
2. If any compound has a %RSD $>30\%$ or a %D $>25\%$ for volatiles and $>30\%$ for semivolatiles:
 - a. Flag positive results for that compound as estimate (J).
 - b. Flag non-detects for that compound as estimated (UJ) if the %RSD or %D is $>50\%$.

A separate worksheet should be filled out for each initial curve.

REGION I
Data Review Worksheet

IV B. SEMIVOLATILE CALIBRATION VERIFICATION

Date of Initial Calibration :
Dates of Continuing Calibrations :
Instrument ID :

<u>DATE</u>	<u>CRITERIA OUT</u> RF,%RSD,RF,%D	<u>COMPOUND (VALUE)</u>
-------------	--------------------------------------	-------------------------

_____	Samples Affected:	
-------	-------------------	--

_____	Samples Affected:	
-------	-------------------	--

_____	Samples Affected:	
-------	-------------------	--

_____	Samples Affected:	
-------	-------------------	--

_____	Samples Affected:	
-------	-------------------	--

_____	Samples Affected:	
-------	-------------------	--

_____	Samples Affected:	
-------	-------------------	--

_____	Samples Affected:	
-------	-------------------	--

_____	Samples Affected:	
-------	-------------------	--

_____	Samples Affected:	
-------	-------------------	--

_____	Samples Affected:	
-------	-------------------	--

_____	Samples Affected:	
-------	-------------------	--

_____	Samples Affected:	
-------	-------------------	--

_____	Samples Affected:	
-------	-------------------	--

See worksheet IV-A for criteria and actions.

A new worksheet should be filled out for each initial curve.

REGION I
Data Review Worksheet

V A. BLANK ANALYSIS RESULTS (Sections 1 & 2)

List the contamination in the blanks below.

1. Laboratory Blanks

CONCENTRATION LEVEL: _____ (low or medium)*

<u>DATE</u>	<u>LAB ID</u>	<u>FRACTION/ MATRIX</u>	<u>COMPOUND</u>	<u>CONCENTRATION/ UNITS</u>
-------------	---------------	-----------------------------	-----------------	---------------------------------

2. Equipment (Field) and Trip Blanks

<u>DATE</u>	<u>LAB ID</u>	<u>FRACTION/ MATRIX</u>	<u>COMPOUND</u>	<u>CONCENTRATION/ UNITS</u>
-------------	---------------	-----------------------------	-----------------	---------------------------------

*A separate worksheet should be used for low and medium level blanks.

V B. BLANK ANALYSIS RESULTS (Section 3)

3. Blank actions

Action levels should be based upon the highest concentration of contaminant determined in any blank. The action level for samples which have been concentrated or diluted should be multiplied by the concentration/dilution factor. No positive sample result should be reported unless the concentration of the compound in the sample exceeds the action level of 10 x's the amount for any other compound. Specific actions are as follows:

1. The concentration is less than the CRQL, report the CRQL.
2. The concentration is greater than the CRQL, but less than the action level, report the concentration found U.
3. The concentration is greater than the action level, report the concentration unqualified.

For examples refer to the Regional Guidelines.

Common contaminants = methylene chloride, acetone, 2-butanone, toluene, and base-neutral fraction phthalate ester compounds (i.e., bis(2-ethyl hexyl)phthalate.

LEVEL: _____

<u>COMPOUND</u>	<u>MAX. CONC./</u> <u>UNITS</u>	<u>ACTION LEVEL/</u> <u>UNITS</u>	<u>CRQL</u>
-----------------	------------------------------------	--------------------------------------	-------------

A separate worksheet should be used for low and medium level blanks.

REGION I
Data Review Worksheets

VI. SURROGATE SPIKE RECOVERIES

List the percent recoveries which do not meet the criteria for surrogate recovery.

Matrix: _____

TR #'S	TOL	<u>VOA</u> 4-BFB	DCE	NBZ	<u>B/N</u> FBP	TPH	PHL	<u>A</u> 2FP	TBP	<u>PEST</u> DBC*
<u>QC Limits</u>	<u>88</u>	<u>86</u>	<u>76</u>	<u>35</u>	<u>43</u>	<u>33</u>	<u>30</u>	<u>21</u>	<u>10</u>	<u>24</u>
	to	to	to	to	to	to	to	to	to	to
<u>(WATERS)</u>	<u>110</u>	<u>115</u>	<u>114</u>	<u>114</u>	<u>116</u>	<u>141</u>	<u>94</u>	<u>100</u>	<u>123</u>	<u>154</u>

<u>(SOLIDS)</u>	<u>84</u>	<u>59</u>	<u>70</u>	<u>23</u>	<u>30</u>	<u>18</u>	<u>24</u>	<u>25</u>	<u>19</u>	<u>20</u>
	to	to	to	to	to	to	to	to	to	to
	<u>138</u>	<u>113</u>	<u>121</u>	<u>120</u>	<u>115</u>	<u>137</u>	<u>113</u>	<u>121</u>	<u>122</u>	<u>150</u>

*The DBC windows are advisory only
Surrogate Actions:

PERCENT RECOVERY

	<u><10%</u>	<u>10%-(MIN)</u>	<u>>R(MAX)</u>
Positive sample results	J	J	J
Non-detected results	R	UJ	A

R(MIN): Denotes lower limit of surrogate recovery range window (i.e., TOL which denotes the VOA surrogate spiking standard toluene -d8) has R(MIN) of 88% for a water matrix sample and a R(MAX) of 110. The acceptance window is therefore 88-110%.

Surrogate action should be applied:

1. If at least two surrogates in a B/N or A fraction or one surrogate in the VOA fraction are out of specification, but have recoveries of >10%.
2. If any one surrogate in a fraction shows <10% recovery.

REGION I
Data Review Worksheets

VII A. MATRIX SPIKE/MATRIX SPIKE DUPLICATE

1. Matrix Spike/Matrix Spike Duplicate Recoveries and Precision

TR Nos. _____, _____ Level: _____ Matrix: _____

List the percent recoveries and RPD's of compounds which do not meet the criteria stated in SW-846. Radian Corporation (the laboratory) must supply the new SW-846 percent recoveries and RPD maximums on a EPA Form III. Contact Radian Corporation (the laboratory) if the revised Form III is not present.

FRACTION/
MS OR MSD

%REC/
COMPOUND

% REC/RPD QC IMIS

QUALIFICATION IS LIMITED TO THE UNSPIKED SAMPLE ONLY.

1. If any compound does not meet the recovery range, follow the actions stated below:

	<u>PERCENT RECOVERY</u>		
	<u><10%</u>	<u>10%-R(MIN)</u>	<u>>R(MAX)</u>
Positive sample results	J	J	J
Non-detected results	R	UJ	A

R(MIN): Denotes lower limit of matrix spike recovery range window (i.e., trichloroethene (water matrix): 71%). R(MAX) for trichloroethene (water matrix) is 120% The acceptance window is therefore 71-120%.

2. If any compound does not meet the RPD criteria, flag positive results for that compound as estimated (J).

A separate worksheet should be used for each MS/MSD pair.

Refer to Appendix A for matrix spike percent RPD maximums and percent recoveries.

REGION I
Data Review Worksheets

VII B. MATRIX SPIKE/MATRIX SPIKE DUPLICATE (Section 2)

3. Matrix Spike Duplicate - Unspiked Compounds

TR Nos. _____, _____

List the concentrations of the unspiked compounds and determine the percent RSD's of the unspiked sample, matrix spike, and matrix spike duplicate. No limits have been developed for the RSD values of the unspiked compounds.

<u>FRACTION</u>	<u>COMPOUND</u>	<u>SAMPLE, MS, MSD CONC.</u>	<u>%RSD</u>
-----------------	-----------------	------------------------------	-------------

The reviewer must use professional judgment to determine if there is a need to qualify any of the unspiked compounds in the sample.

VIII. FIELD DUPLICATE PRECISION

TR Nos. _____, _____ Matrix: _____

List the concentrations of the compounds which do not meet the following RPD criteria:

1. An RPD of <30% for water duplicates.
2. An RPD of <50% for soil duplicates.

<u>FRACTION</u>	<u>COMPOUND</u>	<u>SAMPLE CONC</u>	<u>DUP SAMPLE CONC</u>	<u>RPD</u>
-----------------	-----------------	--------------------	------------------------	------------

ACTIONS:

1. If the results for any compounds do not meet the RPD criteria, flag the positive results for that compound as estimated.
2. If one value is non-detected, and one is above the CRQL:
 - a. Flag the positive result as estimated (J).
 - b. Flag the non-detected result as estimated (UJ).

NOTE: Professional judgment may be utilized to apply duplicate action to all samples of a similar matrix.

A separate worksheet should be filled out for each field duplicate pairs.

REGION I
Data Review Worksheets

IX. INTERNAL STANDARD PERFORMANCE

List the internal standard areas of samples which do not meet the criteria of +100% or -50% of the internal standard area in the associated continuing calibration standard.

<u>SAMPLE ID</u>	<u>DATE</u>	<u>IS OUT</u>	<u>IS AREA/ RT</u>	<u>ACCEPTABLE RANGE</u>	<u>ACTION</u>
------------------	-------------	---------------	------------------------	-------------------------	---------------

ACTION:

1. If an IS area count is outside the criteria -50% or +100% of the associated standard:
 - a. Positive results for compounds quantitated using that IS are flagged as estimated (J) for that sample fraction.
 - b. Non-detects for compounds quantitated using that IS are flagged as estimated (UJ) for that sample fraction.
 - c. If extremely low area counts are reported, or if performance exhibits a major drop-off, then a severe loss of sensitivity is indicated. Non-detects should then be flagged as unusable (R).
2. If an IS retention time varies more than 30 seconds, the chromatographic profile for that sample must be examined to determine if any false positives or negatives exist. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for that sample fraction.

REGION I
Data Review Worksheets

X A. PESTICIDE INSTRUMENT PERFORMANCE (Section 1)

1. DDT Retention Time

List the DDT standards which have a retention time (RT) of less than 12 minutes on the packed column (except OV-1 or OV-101).

<u>SAMPLE ID</u>	<u>DATE</u>	<u>IS OUT</u>	<u>IS AREA/ RT</u>	<u>ACCEPTABLE RANGE</u>	<u>ACTION</u>
------------------	-------------	---------------	------------------------	-------------------------	---------------

Note: A megabore or capillary column standard RT may be shorter than 12 minutes.

ACTION:

If the RT is less than 12 minutes, examine the chromatography to evaluate the separation.
If adequate separation is not achieved, flag all affected compound data as unusable (R).

REGION I
Data Review Worksheets

- X B. PESTICIDE INSTRUMENT PERFORMANCE (Section 2)
 (Form is for both organophosphorous pesticides and herbicides as well as
 organochlorine pesticides)
2. Retention Time Windows

List the compounds which are not within the established windows.

<u>COMPOUND</u>	<u>DATE</u> <u>(TIME)</u>	<u>RT</u>	<u>RT WINDOW</u>	<u>SAMPLES AFFECTED</u>
-----------------	------------------------------	-----------	------------------	-------------------------

Check the sample chromatograms of the samples analyzed after the last in control standard for peaks within an expanded window. If no peaks are present, there is usually no effect on the data. Refer to Regional guidelines for information on qualifying data if peaks are present. If peaks are present, discuss actions below:

REGION I
Data Review Worksheets

X C. PESTICIDE INSTRUMENT PERFORMANCE (Section 3)

3. DDT and Endrin Degradation

List the standards which have a DDT or Endrin breakdown of greater than 20%.

<u>STANDARD ID</u>	<u>DDT OR ENDRIN</u>	<u>PERCENT BREAKDOWN</u>	<u>ENDRIN SAMPLES AFFECTED</u>	<u>DDD, DDE OR ENDRIN KETONE PRESENT</u>
------------------------	--------------------------	------------------------------	------------------------------------	--

If the percent breakdown for DDT is greater than 20%.

1. Flag all positive results for DDT as estimated (J) for all samples following the last in control standard. If no DDT was present, but DDD and/or DDE are positive, then flag the quantification limit for DDT as unusable (R).
2. Flag all positive results for DDD +/- DDE as estimated (J).

If the percent breakdown for Endrin is greater than 20%:

1. Flag all positive results for endrin as estimated (J) for all samples following the last in-control standard. If no endrin was detected, but endrin aldehyde and/or endrin ketone are positive, flag the quantification limit for endrin as unusable (R).
2. Flag all positive results for endrin ketone as estimated (J).

REGION I
Data Review Worksheets

X D. PESTICIDE INSTRUMENT PERFORMANCE (Section 4)

4. DBC Retention Time Check

List the percent difference for the DBC shift greater than 2% for packed columns, greater than 1.5% for wide-bore capillary columns, or greater than 0.3% for narrow-bore capillary columns.

<u>TR #'s</u>	<u>DBC % DIFFERENCE</u>	<u>ACTIONS</u>
---------------	-------------------------	----------------

If the DBC does not meet the retention time criteria, the analysis may be flagged as unusable (R) for the affected samples, but qualification of the data is left up to the professional judgment of the reviewer. Discuss any qualification of the data below:

REGION I
Data Review Worksheets

- XI A. PESTICIDE CALIBRATION (Sections 1 and 2)
 (Form is for both organophosphorous pesticides and herbicides as well as
 organochlorine pesticides.)
1. Initial Calibration

List the compounds which did not meet the Relative Standard Deviation (RSD) criteria of less than 20% for the initial calibration on the quantification column.

<u>DATE</u>	<u>COMPOUND</u>	<u>%RSD</u>	<u>COLUMN</u>	<u>SAMPLES AFFECTED</u>
-------------	-----------------	-------------	---------------	-------------------------

Flag all associated positive results as estimated (J) for samples which did not meet the %RSD criteria.

2. Analytical Sequence

Did the laboratory supply the analytical sequence utilized and the appropriate retention time windows for each analyte in the check standard as per the requirements of Method 8000 in SW-846 3rd Edition and the Radian QAPP. Yes or No.

If no,

The data may be affected. The data reviewer must use professional judgment to determine the severity of the effect and qualify the data accordingly. Discuss any actions below. Refer to Method 8000 protocols for guidance on any action taken. Contact the laboratory to discuss any anomalies encountered to prevent reoccurrence on future analysis.

REGION I
Data Review Worksheets

XI B. PESTICIDE CALIBRATION (Section 3)

3. Continuing Calibration

List the compounds which did not meet the percent difference (%D) criteria of $\pm 15\%$ on the quantification column or $\pm 20\%$ on the confirmation for the continuing calibration.

<u>DATE</u>	<u>COMPOUND</u>	<u>%D</u>	<u>COLUMN</u>	<u>SAMPLES AFFECTED</u>
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

If the %D criteria is not met, flag all associated positive results as estimated (J).

REGION I
Data Review Worksheets

XII. SAMPLE QUANTIFICATION

In the space below, please show a minimum of one sample calculation per fraction:

VOA:

BNA:

PEST/PCB:

Organosporous pesticides

Herbicides

PCDD/PCDF

Organochlorine pesticides

XIII. ADDITIONAL APPENDIX IX Method specific validation checks involving
 Section A: Method 8150 (Herbicides); Section B: Method 8140
 (Organophosphorous Pesticides); Section C: Method 8080 (Organochlorine
 Pesticide and PCBs),

A. Method 8150's surrogate standard is 2,4-dichlorophenyl acetic acid

List the surrogate percent recovery:

List the QC limits listed on the Summary Form for each obtained from the
 Herbicides Table (8150)

Surrogate Actions:

Percent Recovery
<10% 10%-R(MIN)>R(MAX)

Positive Sample Results

J

J

J

Non-detected Results

R

UJ

advisory only

Note: The laboratory must report an acceptable surrogate recovery range as per the
 requirements of Sections 8.3 of Method 8150. As such, the Method 8150 surrogate spike
 acceptance window is 50-150% for both waters and solids. The R(MIN) is 50% and
 R(MAX) is 150%. Method 8150's MS/MSD's are 2,4-D, Dinoseb, 2,4,5-T and 2,4,5-TP
 (Silvex).

List the Method 8150 (Herbicides) percent recoveries and RPDs of compounds which do
 not meet the criteria listed on EPA Form III by Radian (laboratory).

<u>Fraction/ MS or MSD</u>	<u>% Rec Compound</u>	<u>% Rec/RPD</u>	<u>QC Limits</u>
--------------------------------	---------------------------	------------------	------------------

TABLE 8150: HERBICIDES

<u>Parameter</u>	<u>Matrix Spike (a)</u>				<u>Surrogate Spike</u>	
	<u>Precision</u>		<u>Accuracy</u>		<u>Accuracy (c)</u>	
	<u>RPD% (b)</u>		<u>% of Recovery</u>		<u>% of Recovery</u>	
	<u>Water</u>	<u>Solids</u>	<u>Water</u>	<u>Solids</u>	<u>Water</u>	<u>Solids</u>
SW-846 (3rd Ed.) Method 8150						
Herbicides:						
2,4-D	13	N/A	69-159	N/A	--	--
Dinoseb	15	N/A	56-150	N/A	--	--
2,4,5-TP	25	N/A	29-182	N/A	--	--
2,4,5-T	9	N/A	82-138	N/A	--	--
2,4-Dichlorophenylaceticacid	--	--	--	--	50-150	50-150

- (a) Matrix spike precision and accuracy goals, where stated, are found in EPA method references and will be used as starting points. Limits developed in-house will be used and updated throughout the program.
- (b) "RPD" = relative percent difference. Precision is expressed according to the type of measurement (i.e., for field duplicates precision is expressed as the RPD between duplicate results).
- (c) Accuracy goals stated are from EPA CLP, SW-846 methods, or Radian derived limits. These limits will be used as starting points for control chart generation. Limits developed in-house will be used and updated throughout the program.

N/A Not available (to be developed by Radian).

- B. The surrogate standard for Method 8140 is triphenyl phosphate. For waters the acceptance window is 40-140% [R(MIN):40; R(MAX):140] and for solids the acceptance window is 42-154% [R(MIN):42; R(MAX):154]. Method 8140's MS/MSD compounds are: dimethoate, thionazin, sulfotepp, disulfotan, ethylparathion, famphur, methylparathion and phorate. Refer to Table 8140.

List the percent recovery:

List the QC limits listed on the Summary Form for each obtained from the Organophosphorous Pesticides Table (8140)

Surrogate Actions:

Percent Recovery
<10% 10%-R(MIN)>R(MAX)

Positive Sample Results

J

J

J

Non-detected Results

R

UJ

advisory only

List the Method 8140 (Organophosphorous Pesticide) percent recoveries and RPD's of compounds which do not meet the criteria listed on EPA Form III by Radian (laboratory).

Fraction/
MS or MSD

% Rec
Compound

% Rec/RPD

QC Limits

TABLE 8140: ORGANOPHOSPHOROUS PESTICIDES

<u>Parameter</u>	<u>Standard Deviation</u>		<u>Accuracy (a) % of Recovery</u>		<u>Accuracy (a) % of Recovery</u>	
	<u>Water</u>	<u>Solids</u>	<u>Water</u>	<u>Solids</u>	<u>Water</u>	<u>Solids</u>
SW-846 Method 8140						
<u>Organophosphorus Pesticides:</u>						
DELETED → Dimethoate	N/A	N/A	N/A	N/A	--	--
Famphur	50	33	10-168	10-136	--	--
Ethylparathion	14	21	45-112	29-133	--	--
12-90 → Thionazin	N/A	N/A	N/A	N/A	--	--
Disulfoton	36	39	10-138	10-219	--	--
Methyl Parathion	35	36	10-213	10-177	--	--
Phorate	32	33	10-183	10-166	--	--
→ Sulfotepp	N/A	N/A	32-124	10-165	--	--
Triphenylphosphate	--	--	--	--	23-176	28-174

(a) These limits were developed in-house. Radian will update these throughout the program.

→ N/A Not available (to be developed by Radian).

- C. Method 8080 (Organochlorine Pesticide and PCBs)
The surrogate standard is dibutylchlorendate. For waters the acceptance window is 24-154% [R(MIN):24; R(MAX):154] and for solids the acceptance window is 20-150% [R(MIN):20; R(MAX):150].

List the percent recovery:

List the QC limits listed on the Summary Form for each obtained from the Organochlorine Pesticide and PCB Table 8080.

Surrogate Actions:

	<u>Percent Recovery</u>		
	<u><10%</u>	<u>10%-R(MIN)</u>	<u>>R(MAX)</u>
Positive Sample Results	J	J	J
Non-detected Results	R	UJ	advisory only

Note: The laboratory must report an acceptable surrogate recovery range as per the requirements of Section 8.3 of Method 8080. As such, the Method 8080 surrogate spike acceptance window is 24 - 154% for waters and 20 - 150% for solid notices. The MS/MSD compounds are: gamma - BHC, heptochlor, aldrin, dieldrin, endrin and 4,4¹-DDT. Refer to Table 8080.

List the Method 8080 (Organochlorine Pesticide and PCBs) percent recoveries and RPDs of compounds which do not meet the criteria listed on EPA Form III by Radian (laboratory).

If any compound does not meet the RPD criteria, flag positive results for that compound as estimated (J).

<u>Fraction/ MS or MSD</u>	<u>% Rec Compound</u>	<u>% Rec/RPD</u>	<u>QC Limits</u>
--------------------------------	---------------------------	------------------	------------------

TABLE 8080: ORGANOCHLORINE PESTICIDES AND PCBs

<u>Parameter</u>	<u>Matrix Spike (a)</u>				<u>Surrogate Spike</u>	
	<u>Precision</u>		<u>Accuracy</u>		<u>Accuracy (c)</u>	
	<u>RPD% (b)</u>		<u>% of Recovery</u>		<u>% of Recovery</u>	
	<u>Water</u>	<u>Solids</u>	<u>Water</u>	<u>Solids</u>	<u>Water</u>	<u>Solids</u>
<u>Pesticides/Polychlorinated</u>						
<u>Biphenyls:</u>						
gamma-BHC	15	50	56-123	46-127	--	--
Heptachlor	20	31	40-131	35-130	--	--
Aldrin	22	43	40-120	34-132	--	--
Dieldrin	18	38	52-126	31-134	--	--
Endrin	21	45	56-121	42-139	--	--
4,4'-DDT	27	50	38-127	23-134	--	--
Dibutylchloredate	--	--	--	--	24-154	20-150

- (a) Matrix spike precision and accuracy goals, where stated, are found in EPA method references and will be used as starting points. Limits developed in-house will be used and updated throughout the program.
- (b) "RPD" = relative percent difference. Precision is expressed according to the type of measurement (i.e., for field duplicates precision is expressed as the RPD between duplicate results).
- (c) Accuracy goals stated are from EPA CLP, SW-846 methods, or Radian derived limits. These limits will be used as starting points for control chart generation. Limits developed in-house will be used and updated throughout the program.

XIV.Dioxin (Method 8280: Full Scan Tetra-Octa including 2,3,7,8-TCDD)

Refer to Appendix B for Radian Dioxin Summary Forms. QAPP QA/QC criteria for dioxins are listed below:

**PCDD AND PCDF (DIOXINS AND FURANS)
QUALITY CONTROL OBJECTIVES**

Parameter	Precision RPD % for Duplicate Analyses (a) Water
PCDD	60-140
PCDF	60-140

(a) These objectives are for recovery check samples, it is anticipated that the field samples will fall within these objectives.

ORGANIC REGIONAL DATA ASSESSMENT

REFERENCE NO.: SITE: CIBA-GEIGY, CRANSTON, RI
 LABORATORY: NO. OF SAMPLES/ MATRIX:
 SDG #: REVIEWER (IF NOT ESD):
 SW846, 3RD EDITION: REVIEWER'S NAME:
 COMPLETION DATE:

DATA ASSESSMENT SUMMARY

	<u>VOA</u>	<u>BNA</u>	<u>PEST</u>	<u>Organo-P,CL PESTICIDES</u>	<u>HERBICIDES</u>	<u>PCDD PCDF</u>
1. HOLDING TIMES	_____	_____	_____	_____	_____	_____
2. GC/MS TUNE/INSTR. PERF.	_____	_____	_____	_____	_____	_____
3. CALIBRATIONS	_____	_____	_____	_____	_____	_____
4. BLANKS	_____	_____	_____	_____	_____	_____
5. SURROGATES	_____	_____	_____	_____	_____	_____
6. MATRIX SPIKE/DUP	_____	_____	_____	_____	_____	_____
7. OTHER QC	_____	_____	_____	_____	_____	_____
8. INTERNAL STANDARDS	_____	_____	_____	_____	_____	_____
9. COMPOUND IDENTIFICATION	_____	_____	_____	_____	_____	_____
10. SYSTEM PERFORMANCE	_____	_____	_____	_____	_____	_____
11. OVERALL ASSESSMENT	_____	_____	_____	_____	_____	_____

O = Data had no problems/or qualified due to minor problems.

M = Data qualified due to major problems.

Z = Data unacceptable.

X = Problems, but do not affect data.

ACTIONS ITEMS:

AREAS OF CONCERN:

APPENDIX A
MATRIX SPIKE RPD MAXIMUMS
AND PERCENT RECOVERIES

**MS/MSD QA/QC CRITERIA
VOLATILES**

Matrix spike(a)				
<u>Parameter</u>	Precision		Accuracy (a)	
	RPD %(b)		% of Recovery	
	<u>Water</u>	<u>Solids</u>	<u>Water</u>	<u>Solids</u>
<u>Volatiles</u>				
Trichloroethene	14	24	71-120	62-137
Benzene	11	21	76-127	66-142
Toluene	13	21	76-125	59-139
Chlorobenzene	13	21	75-130	60-133
1,1-Dichloroethene	14	22	61-145	59-172
Toluene-d8	--	--	--	--
4-Bromofluorobenzene	--	--	--	--
1,2-Dichloroethane-d4	--	--	--	--

- (a) Matrix spike precision and accuracy goals, where stated, are found in EPA method references and will be used as starting points. Limits developed in-house will be used and updated throughout the program.
- (b) "RPD" = relative percent difference. Precision is expressed according to the type of measurement (i.e., for field duplicates precision is expressed as the RPD between duplicate results).
- (c) Surrogate recoveries are listed on page 9 of 28.

**MS/MSD QA/QC CRITERIA
SEMIVOLATILES**

Matrix spike(a)				
<u>Parameter</u>	Precision RPD %(b)		Accuracy (a) % of Recovery	
	<u>Water</u>	<u>Solids</u>	<u>Water</u>	<u>Solids</u>
<u>GC/MS Semivolatiles:</u>				
Phenol	42	35	12-89	26-90
2-Chlorophenol	40	50	27-123	25-102
1,4-Dichlorobenzene	28	27	36-97	28-104
N-Nitroso-di-n-propyl-amine	38	38	41-116	41-126
1,2,4-Trichlorobenzene	28	23	39-98	38-107
Acenaphthylene	31	39	46-118	31-137
4-Nitrophenol	50	50	10-80	11-114
2,4-Dinitrotoluene	38	47	24-96	28-89
Pentachlorophenol	50	47	9-103	17-109
Pyrene	31	36	26-127	35-142
Nitrobenzene-d5	--	--	--	--
2-Fluorobiphenyl	--	--	--	--
p-Terphenyl-d14	--	--	--	--
2-Fluorophenol	--	--	--	--
2,4,6-Tribromophenol	--	--	--	--
Propazine	18*	5*	34-118*	77-100*
Tinuvin-327	15*	6*	47-123*	77-107*

- (a) Matrix spike precision and accuracy goals, where stated, are found in EPA method references and will be used as starting points. Limits developed in-house will be used and updated throughout the program.
- (b) "RPD" = relative percent difference. Precision is expressed according to the type of measurement (i.e., for field duplicates precision is expressed as the RPD between duplicate results).
- (c) Surrogate recoveries are listed on page 9 of 28.

* These values were calculated based on data collected from the reagent spike blanks and fine sediment samples. These data will be updated as more data points become available throughout Round I.

**MS/MSD QA/QC CRITERIA
PESTICIDE/PCBs**

Matrix spike(a)				
<u>Parameter</u>	Precision RPD %(b)		Accuracy (a) % of Recovery	
	<u>Water</u>	<u>Solids</u>	<u>Water</u>	<u>Solids</u>
<u>Pesticides/Polychlorinated</u>				
<u>Biphenyls:</u>				
gamma-BHC	15	50	56-123	46-127
Heptachlor	20	31	40-131	35-130
Aldrin	22	43	40-120	34-132
Dieldrin	18	38	52-126	31-134
Endrin	21	45	56-121	42-139
4,4'-DDT	27	50	38-127	23-134
Dibutylchloredate	--	--	--	--

- (a) Matrix spike precision and accuracy goals, where stated, are found in EPA method references and will be used as starting points. Limits developed in-house will be used and updated throughout the program.
- (b) "RPD" = relative percent difference. Precision is expressed according to the type of measurement (i.e., for field duplicates precision is expressed as the RPD between duplicate results).
- (c) Surrogate recoveries are listed on page 9 of 28.

APPENDIX B
DIOXIN SUMMARY FORMS
METHOD 8280 (FULL-SCAN)
TETRA - OCTA

WORKSHEET TO CALCULATE TOXICITY EQUIVALENT FACTORS (TEFs)

INSTRUCTIONS: TEFs BASED ON THE DRAFT 4/89 CLP DIOXIN PROTOCOL

Results for Each Sample Are Given Two Lines: The top line is the concentration of the analyte in ppb.

The bottom line equates that concentration to the toxic equivalent of 2,3,7,8-TCDD.

[illegible]

SW-846 METHOD 8280

DIOXIN

SUMMARY

FORMS

**CIBA-GEIGY SITE
CRANSTON, RHODE ISLAND**

Form 2: Internal Standard Recovery
Form 3: Initial Calibration Summary
Form 4: Continuing Calibration Summary
Form 5: Standard Worksheet
Form 6: Sample Worksheet
Form 7: Quality Control Report
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Form B-6A: Dioxin Raw Sample Data (PCDD, PCDF)
Form B-6B: Dioxin Raw Sample Data (HxCDD, HxCDF)
Form B-6C: Dioxin Raw Sample Data (HpCDD, HpCDF)
Form B-6D: Dioxin Raw Sample Data (OCDD, OCDF)

DIOXIN AND FURAN DATA VALIDATION WORKSHEET

Based on Method 8280 in SW-846 (3rd Edition) and Radian's Standard Operating Procedure
11/09/90

Prepared by Diana Baldi, CIBA-GEIGY

Reviewed by John Lorenzo, Woodward-Clyde Consultants

REQUIREMENTS	ACCEPTABLE LIMITS	ACTION IF EXCEEDED
Initial calibration 5 point curve in triplicate (Form 3)	% RSD between 3 runs must be <15% for all analytes	Recalculate concentration using daily RF; note in narrative; request reanalysis for compliance
PCDD/PCDF retention time standard	First and last eluting isomer must be detected	Request reanalysis
Chromatography standard (included in RT standard)	>25% valley between 1,2,3,4- TCDD and 2,3,7,8-TCDD	Look for high bias in any positives for 2,3,7,8-TCDD; if sample results affected, request reanalysis
Continuing calibration (Form 4)	a) RF <30% of eve RF from initial calibration b) All major chlorine isotope cluster abundances within 15% of theoretical	a) Recalculate concentration using daily RF; note in narrative b) Look for false negatives due to failing ion criteria; <u>Note:</u> Calculate EMPC, include EMPC in TE; if action level request reanalysis
Identification of positives	a) Major chlorine isotope cluster ions must be within 15% of theoretical b) COCL loss peak must be present c) Peak maxima must agree within 2 scans	a) See b) above b) Look for false positives c) Look for false positives
Internal standards (Form 2)	a) Must be present at least 10:1 signal to noise ratio in all standards and samples b) Should have absolute recovery greater than 40%	a) Require reanalysis b) No action if S:N criteria met
Surrogate accuracy	Within 40% to 120%	Options: a) No action b) Add 'J' qualifier to associated homologs

The process of data validation flows as follows:

Step 1: Performance evaluation (PE) samples will be incorporated in the field shipment of samples to Radian. The results of the PE samples will be validated according to the protocols stated above. The validated results will be called in to USEPA Region I designee. Upon reporting the results, the true values will be released to CIBA-GEIGY designee (Diana Baldi). If the results are outside the USEPA acceptance limits, a reanalysis of the entire sample set will be required. If the results of the PE are acceptable, then validation of the remaining samples will proceed.

Note: The worksheets provided as an appendix will be used when appropriate.

Step 2: Verify all internal standards in all samples have S:N 10:1 utilizing Form 2.

Step 3: Calculate all ion ratios (Form 5) for peaks that may be positives in samples; spot check ion ratios in the continuing and initial calibration focusing on those congeners that may have false negatives in samples due to ion ratios.

Step 4: Spot check the criteria in the initial and continuing calibration data submitted in Forms 3 and 4. Note if RT for the internal standards shift during the time of analysis for the sample group.

Step 5: Look for RT cutoff points for each homolog based on RT standard. Look for potential false negatives due to being outside the established RT window because of a RT shift.

Step 6: Recalculate all positives and EMPC using either the initial eve RF or the continuing calibration RF (as stated in the above criteria table).

Step 7: Enter the data into the TEF spreadsheet. Look for anything unusual and recheck all entries.

The final results are to be presented in a table that includes the concentration of each of the twelve reported values (2,3,7,8-TCDD, 2,3,7,8-TCDF, tetra thru octa chlorinated dibenzo-p-dioxins and dibenzofurans). The table will also calculate the total Toxicity Equivalents (TE). The TE values are based on the 9 November 1988 Revised Dioxin TE Factors memo.

INTERNAL STANDARD RECOVERY

- *C-TCDD :Carbon 13 labeled 2,3,7,8-tetrachlorodibenzodioxin
- *C-HxCDD:Carbon 13 labeled 1,2,3,6,7,8-hexachlorodibenzodioxin
- *C-OCDD :Carbon 13 labeled octachlorodibenzodioxin
- *C-TCDF :Carbon 13 labeled 2,3,7,8-tetrachlorodibenzofuran
- *C-HpCDF:Carbon 13 labeled 1,2,3,4,6,7,8-heptachlorodibenzofuran

*C-HpCDF: Carbon 13 labeled 1,2,3,4,6,7,8-heptachlorodibenzofuran

INSTRUMENT
CONC.
TIME
STD.ID.

RF

SD

RSD

2378 TCDD
12378 PeCDD
123478 HxCDD
123678 HxCDD
123789 HxCDD
1234678 HpCDD
OCDD
2378 TCDF
12378 PeCDF
23478 PeCDF
123478 HxCDF
123678 HxCDF
123789 HxCDF
234678 HxCDF
1234678 HpCDF
1234789 HpCDF
OCDF

[illegible]

- *C-TCDD¹
- *C-TCDF¹
- *C-HxCDD²
- *C-HpCDF²
- *C-OCDD²

conc. pg/ul

*C-TCDD	*13C12-2,3,7,8-TCDD	
*C-TCDF	*13C12-2,3,7,8-TCDF	
*C-HxCDD	*13C12-1,2,3,6,7,8-HxCDD	
*C-HpCDF	*13C12-1,2,3,4,6,7,8-HpCDD	
*C-OCDD	*13C12-OCDDP	

1. Based on recovery standard 13C-1234-TCDD
2. Based on recovery standard 12C-123789-HxCDD

FORM 4
CONTINUING CALIBRATION SUMMARY

INSTRUMENT

		INITIAL CURVE (RF)	CONT. CALIB. (RF)	RPD	CONT. CALIB. (RF)	RPD	CONT. CALIB. (RF)	RPD
CONC. DATE/TIME STD. ID.								
2378	TCDD							
12378	PeCDD							
123478	HxCDD							
123678	HxCDD							
123789	HxCDD							
1234678	HpCDD							
	OCDD							
2378	TCDF							
12378	PeCDF							
23478	PeCDF							
123478	HxCDF							
123678	HxCDF							
123789	HxCDF							
234678	HxCDF							
1234678	HpCDF							
1234789	HpCDF							
	OCDF							
*C-TCDD ¹								
*C-TCDF ¹								
*C-HxCDD ²								
*C-HpCDF ²								
*C-OCDD ²								

**FORM 5
STANDARD WORKSHEET**

DATE : _____
 INJ TIME: _____
 STD ID : _____
 COLUMN : _____
 INST ID : _____

	SCAN# 'E	ION	AREA	ION	AREA*	AREA/AREA*	CONC	REF
13C-1234-TCDD	_____	(332)	_____	(334)	_____	_____	_____	_____
13C-2378-TCDD**	_____	(332)	_____	(334)	_____	_____	_____	_____
2378-TCDD ¹	_____	(320)	_____	(322)	_____	_____	_____	_____
12378-PeCDD ¹	_____	(358)	_____	(356)	_____	_____	_____	_____
13C-123789-HxCDD	_____	(404)	_____	(402)	_____	_____	_____	_____
13C-123678-HxCDD***	_____	(404)	_____	(402)	_____	_____	_____	_____
__2378-HxCDD ²	_____	(392)	_____	(390)	_____	_____	_____	_____
1234678-HpCDD ²	_____	(426)	_____	(424)	_____	_____	_____	_____
13C-OCDD***	_____	(470)	_____	(472)	_____	_____	_____	_____
OCDD ³	_____	(458)	_____	(460)	_____	_____	_____	_____
13C-2378-TCDF**	_____	(316)	_____	(318)	_____	_____	_____	_____
2378-TCDF ⁴	_____	(304)	_____	(306)	_____	_____	_____	_____
_2378-PeCDF ⁴	_____	(342)	_____	(340)	_____	_____	_____	_____
_2378-HxCDF ⁵	_____	(376)	_____	(374)	_____	_____	_____	_____
13C-1234678-HpCDF***	_____	(422)	_____	(420)	_____	_____	_____	_____
__2378-HpCDF ⁵	_____	(410)	_____	(408)	_____	_____	_____	_____
OCDF ⁵	_____	(442)	_____	(444)	_____	_____	_____	_____

- * - Ion used for quantitation
- ** - Quantitation based on 13C-1,2,3,4-TCDD
- *** - Quantitation based on 13C-1,2,3,7,8,9-HxCDD
- 1 - Int Std 13C-2,3,7,8-TCDD
- 2 - Int Std 13C-1,2,3,6,7,8-HxCDD
- 3 - Int Std 13C-OCDD
- 4 - Int Std 13C-2,3,7,8-TCDF
- 5 - Int Std 13C-1,2,3,4,6,7,8-HpCDF

NOTE: If more than one ratio is required please write ratios directly on EICP.

**FORM 6
SAMPLE WORKSHEET**

DATE : _____
 INJ TIME : _____
 CLIENT ID : _____
 SAMPLE ID : _____
 SAMPLE SIZE: _____

COLUMN : _____
 INST ID : _____
 CURVE/STDS: _____
 COMMENTS : _____

	SCAN# 'S	ION	AREA	ION	AREA*	AREA/AREA*	CONC	RRF	DL
13C-1234-TCDD	_____	(332)	_____	(334)	_____	_____	_____	_____	_____
13C-2378-TCDD**	_____	(332)	_____	(334)	_____	_____	_____	_____	_____
2378-TCDD ¹	_____	(320)	_____	(322)	_____	_____	_____	_____	_____
TOTAL-TCDD ¹	_____	(320)	_____	(322)	_____	_____	_____	_____	_____
12378-PeCDD ¹	_____	(358)	_____	(356)	_____	_____	_____	_____	_____
TOTAL-PeCDD ¹	_____	(358)	_____	(356)	_____	_____	_____	_____	_____
13C-123789-HxCDD	_____	(404)	_____	(402)	_____	_____	_____	_____	_____
13C-123678-HxCDD***	_____	(404)	_____	(402)	_____	_____	_____	_____	_____
XX2378-HxCDD ²	_____	(392)	_____	(390)	_____	_____	_____	_____	_____
TOTAL-HxCDD ²	_____	(392)	_____	(390)	_____	_____	_____	_____	_____
1234678-HpCDD ²	_____	(426)	_____	(424)	_____	_____	_____	_____	_____
TOTAL-HpCDD ²	_____	(426)	_____	(424)	_____	_____	_____	_____	_____
13C-OCDD***	_____	(470)	_____	(472)	_____	_____	_____	_____	_____
OCDD ³	_____	(458)	_____	(460)	_____	_____	_____	_____	_____
13C-2378-TCDF**	_____	(316)	_____	(318)	_____	_____	_____	_____	_____
2378-TCDF ⁴	_____	(304)	_____	(306)	_____	_____	_____	_____	_____
TOTAL-TCDF ⁴	_____	(304)	_____	(306)	_____	_____	_____	_____	_____
X2378-PeCDF ⁴	_____	(342)	_____	(340)	_____	_____	_____	_____	_____
TOTAL-PeCDF ⁴	_____	(342)	_____	(340)	_____	_____	_____	_____	_____
XX2378-HxCDF ⁵	_____	(376)	_____	(374)	_____	_____	_____	_____	_____
TOTAL-HxCDF ⁵	_____	(376)	_____	(374)	_____	_____	_____	_____	_____
13C-1234678-HpCDF***	_____	(422)	_____	(420)	_____	_____	_____	_____	_____
XXX2378-HpCDF ⁵	_____	(410)	_____	(408)	_____	_____	_____	_____	_____
TOTAL-HpCDF ⁵	_____	(410)	_____	(408)	_____	_____	_____	_____	_____
OCDF ⁵	_____	(442)	_____	(444)	_____	_____	_____	_____	_____

- * - Ion used for quantitation
- ** - Quantitation based on 13C-1,2,3,4-TCDD
- *** - Quantitation based on 13C-1,2,3,7,8,9-HxCDD
- 1 - Int Std 13C-2,3,7,8-TCDD
- 2 - Int Std 13C-1,2,3,6,7,8-HxCDD
- 3 - Int Std 13C-OCDD
- 4 - Int Std 13C-2,3,7,8-TCDF
- 5 - Int Std 13C-1,2,3,4,6,7,8-HpCDF

TEF _____

NOTE: If more than one ratio is required please write ratios directly on EIC

FORM 7
QUALITY CONTROL REPORT

CASE NO. _____
SITE _____
EPA SAMPLE ID. _____

MATRIX SPIKE/MATRIX SPIKE DUPLICATE RESULTS

COMPOUND	SAMPLE CONC.	SPIKE ADDED MS	MS CONC.	MS %REC	SPIKE ADDED MSD	MSD CONC.	MSD %REC.	%RPD
(units)	()	()	()		()	()		
TCDD								
PeCDD								
HxCDD								
HpCDD								
OCDD								
TCDF								
PeCDD								
HxCDD								
HpCDD								
OCDD								

Sample No.: _____

Analyst(s): _____

TCDD Required 320/322 Ratio Window is 0.65-0.89

No. Peaks	Scan No.		Confirm as TCDD Y/N	Quantitated vs C-13 2378-TCDD	C-13 1234-TCDD	Dilution	Conc.
		320/322					
						Total TCDD:	

TCDF Required 304/306 Ratio Window is 0.65-0.89

No. Peaks	Scan No.	304/306	Con-	Quantitated vs		Dilution	Conc.
			firmed as TCDD L/E	C-13 2378-TCDD	C-13 1234-TCDD		
			TCDD		1234-TCDD		
Total TCDF:							

Sample No. : _____

Analyst(s): _____

No. Peaks	Scan No.		Confirm as PCDD Y/N	Quantitated vs C-13 2378-TCDD	C-13 1234-TCDD	Dilution	Conc.
		358/356					
						Total PeCDD:	

No. Peaks	Scan No.		Con- firmed as PCDD Y/N	Quantitated vs C-13 237B-TCDD	C-13 1234-TCDD	Dilution	Conc.
	342/340						
						Total PeCDF:	

DIOXIN RAW SAMPLE DATA

Sample No. : _____

Analyst(s): _____

[illegible][illegible]

$\{0, \dots, n\} \times \{0, \dots, m\}$

Sample No. : _____

Analyst(s): _____

No. Peaks	Scan No.		Confirm as HpCDD Y/N	Quantitated vs C-13 OCDD	C-13 1234-TCDD	Dilution	Conc.
		426/424					
							Total HpCDD:

No. Peaks	Scan No.		Confirmed as HpCDF Y/N	Quantitated vs C-13 OCDD	C-13 1234-TCDD	Dilution	Conc.
		410/408					
							Total HpCDF:

Sample No. : _____

Analyst(s): _____

No. Peaks	Scan No.		Confirm as OCDD Y/N	Quantitated vs C-13 OCDD	C-13 1234-TCDD	Dilution	Conc.
		458/460					
							Total OCDD:

No. Peaks	Scan No.		Confirmed as OCDD Y/N	Quantitated vs C-13 OCDD	C-13 1234-TCDD	Dilution	Conc.
		442/444					
							Total OCDF: